



Prepared for:  
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## FIELD SAMPLING PLAN

## REMEDIAL DESIGN

Madison County Mines OU5 Site  
Madison County, Missouri

November 2013

EPA Contract No.: EP-S7-05-06  
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ENERGY WATER INFORMATION GOVERNMENT

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## Abbreviations and Acronyms

AES	Architect/Engineering Services
BVSPC	Black & Veatch Special Projects Corp.
CERCLIS	Comprehensive Environmental Response, Compensation, and Liability Information System
CFR	Code of Federal Regulations
COC	Chain of Custody
DQOs	Data Quality Objectives
EPA	U.S. Environmental Protection Agency
FSP	Field Sampling Plan
GPS	Global Positioning System
HPLC	High Performance Liquid Chromatography
HSP	Health & Safety Plan
IDW	Investigation-derived waste
LSFR	Little St. Francis River
MCL	Maximum Contaminant Levels
mg/kg	Milligram per Kilogram
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NPL	National Priorities List
OU	Operable Unit
PPE	Personal Protective Equipment
PRG	Preliminary Remediation Goal
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
RD	Remedial Design
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
ROD	Record of Decision
SOPs	Standard Operating Procedures
TAL	Target Analyte List
TCLP	Toxicity Characteristic Leaching Procedure
USDOT	U.S. Department of Transportation
WQS	Water Quality Standards
WP	Work Plan
XRF	X-ray Fluorescence Spectroscopy



## **1.0 Introduction**

The U.S. Environmental Protection Agency (EPA) has initiated a Remedial Design (RD) of Madison County Mines Operable Unit (OU) 5 Site near Fredericktown in southeastern Missouri. Documents detailing the methods that will guide RD activities at the Site consist of a work plan (WP), this field sampling plan (FSP), a quality assurance project plan (QAPP), and a health and safety plan (HSP).

This FSP presents a detailed description of activities planned for the RD activities at the Site. These RD activities will include collection of:

- Surface and subsurface samples from chat piles and other mining waste areas around the Catherine and Skaggs sub sites.
- Surface water and sediment samples from the Logtown Branch and two (2) unnamed intermittent streams that flow from the Catherine and Skaggs sub sites.
- Floodplain surface and subsurface soil samples along the Logtown Branch and two (2) unnamed intermittent streams that flow from the Catherine and Skaggs sub sites.
- Groundwater samples from eight (8) monitoring wells that will be installed as part of this RD.

In addition to describing these activities, this RD-FSP describes the operational history and current state of the Site, the sample numbering system that will be employed during RD activities, sample collection and equipment decontamination procedures, management of investigation-derived wastes, and methods to document these activities.

Section 1.2 of this FSP describes the operational history of the Catherine and Skaggs sub sites (OU5). Section 1.3 briefly describes previous investigations at each sub site. Section 2 summarizes the objectives of this investigation which are presented in more detail in the accompanying QAPP (BVSPC 2013). Section 3 describes the sampling and analysis that will be performed at the Site. Section 4 describes the sample numbering system, Section 5 describes sample decontamination procedures, Section 6 describes management of investigation-derived wastes (IDW), and Section 7 describes the documentation requirements for this effort.

This FSP has been prepared by Black & Veatch Special Projects Corp. (BVSPC) under U.S. EPA Contract EP-S7-05-06, Task Order 126.

### **1.1 Site Location and Background**

The Madison County Mines Site, Comprehensive Environmental Response, Compensation, and Liability Information System (CERCLIS) identification number

MOD098633415, is located near Fredericktown in southeastern Missouri. The Site is located at the southern end of the Old Lead Belt where heavy metal mining has occurred since the early 1700s. The Site is located about 80 miles south of St. Louis, Missouri, on the southeastern edge of the Ozark Uplift (Figure 1-1). Past mining operations have left at least 13 identified major mine waste areas, in the form of tailings and chat deposits, from smelting and mineral processing operations in Madison County. Chat deposits include sand to gravel sized material resulting from the crushing, grinding, and dry separation of the ore material. Tailings deposits include sand and silt sized material resulting from the wet washing or floatation separation of the ore material. The mine waste contains elevated levels of lead and other heavy metals which pose a threat to human health and the environment. The Madison County Mines Site was placed on the National Priorities List (NPL) on September 29, 2003. A more detailed description of the Catherine and Skaggs sub sites can be found below.

## **1.2 Operational History and Present Condition of Mining and Processing Sites**

Lead ore was discovered in the area of Mine La Motte (northwest Fredericktown) by French explorers around 1715 (Hall, 1939). The area was already known to and likely was being exploited by local natives. Mining commenced in the early 1720s and continued intermittently on a comparatively small basis through the 18th century (Shoemaker, date unknown). Mining and beneficiation activities increased significantly at Mine La Motte and what is now known as the Madison Mine beginning in the mid-1840s and expanded throughout Madison County in the period following the Civil War, when the diamond drill became available as an exploration tool (Anonymous). Most of the smaller mines that are the subject of these sampling activities were likely located and operated at this time. Mining in Madison County has produced copper, lead, cobalt, nickel, iron, and small amounts of zinc, silver, and tungsten (Tolman, 1933; James, 1949; USGS et al., 1967). Ore crushing and beneficiation activities took place at many sites in Madison County, including the Catherine Mine and Skaggs sub sites.

Property ownership is taken from the most recent GIS database available, provided by Intrinsicorp (Madison County GIS database, <http://madison.villagis.net>, 2013), for the area and was supplemented with research conducted during the reconnaissance.

### **1.2.1 Catherine Mine Site**

The Catherine Mine sub site is located on the west side of Highway H, about 2 miles northwest of Fredericktown in Section 2, Township 33N, Range 6E (Figures 1-2 and 1-3). The sub site is about 2,500 feet north of the intersection of Highways H and 67 and is accessed from

Highway H.

The Catherine sub site presently consists of remnant chat piles, most of which have been excavated and removed, and a 3.5 acre pond that receives runoff and drainage from the chat area. Surface runoff from the northern part of the site drains northeastward along perennially flowing Logtown Branch, a tributary to the Little St. Francis River (LSFR) (Figure 1-4). Runoff from the southern part of the site flows through an unnamed tributary to Plum Creek, a tributary to the LSFR (Figure 1-6).

Much of the former chat disposal area is now covered with soil, either lead-contaminated or quarry overburden. Chat was identified extending into a wooded area west of the chat disposal area; this chat is covered with a few inches of forest duff and soil. Chat is also exposed along the southern shore of the settling pond on the north side of the chat piles. It is unknown if the entire pond is underlain by chat. Observations of the surface drainages show that both Logtown Branch and the unnamed Plum Creek tributary are free of visible chat and tailings.

Chat at the Catherine sub site consists of angular fragments of crystalline dolomite and silica, ranging from fine sand to fragments of 10 mm, with occasional sand- and gravel-sized fragments. The chat, which has a light gray to light brown color, contains less than 1 percent black, metallic minerals. In general, the material is moderately sorted and matrix supported.

### **1.2.2 Skaggs Mine Site**

The Skaggs Mine sub site is located near the intersection of Highways H and 67, about 2 miles northwest of Fredericktown in Section 2, T33N, R6E (Figures 1-2 and 1-3). The sub site is about a quarter mile south of the Catherine Mine sub site and possibly was part of original Catherine Mine sub site. A portion of the Skaggs sub site located west of Highway H and is accessed from Highway H.

The Skaggs sub site presently consists of remnant chat piles, most of which have been excavated and removed, piles of sawdust that locally cover chat, an active garage/shop area, a small scrap yard, and a few miscellaneous buildings that are not in use. The western chat pile is estimated to be 3.1 acres, the central chat pile is estimated to be 8.7 acres, and the eastern chat pile is estimated to be 0.36 acres. Runoff from the western portion of the site drains southwestward through an ephemeral drainage to the unnamed Plum Creek tributary that flows south from the Catherine sub site, and thence to the LSFR (Figure 1-6). Runoff from the eastern portion of the site drains eastward through an unnamed intermittent stream that discharges to the LSFR (Figure 1-5).

Chat was identified extending westward along the base of the sawdust piles into a grassy meadow where it is covered with a few inches of soil. Barren chat is exposed throughout most of the central and northern Skaggs sub site at depths ranging from a few inches to a few feet and the southern margin of the chat extends into a wooded area. Observations of the surface

drainages show that both the western ephemeral drainage and the eastern intermittent drainage contain visible chat and tailings in the areas from which chat was excavated; downstream of the historic piles, the streams are generally free of chat.

Chat at the Skaggs sub site consists of angular fragments of crystalline dolomite and silica, ranging from fine sand to fragments of 10 mm, with occasional sand and gravel-sized fragments. The chat, which has a light gray to light brown color, contains less than 1 percent black, metallic minerals. In general, the material is moderately sorted and matrix supported.

## **1.3 Previous Site Investigations**

### **1.3.1 Catherine Sub Site**

A remedial investigation (RI) report for OU5 was completed in 2008 (BVSPC, 2008). Based on the limited amount of borings installed at the Catherine sub site, it is estimated that approximately 44,000 cubic yards of chat were present at the time of the RI. EPA contractors subsequently transported approximately 205,000 cubic yards of lead impacted soil and chat that was removed from residential property to the Catherine sub site (EPA 2010a). The level of certainty in the estimated volume of chat is low because of the excavated soil that has been placed at the site and additional investigations are necessary to collect information that can be used to provide a more reliable estimate of the volume of chat at the site.

During the RI, continuous 5-foot core samples through the chat were collected at three locations using a track-mounted geoprobe rig. The chat samples were analyzed for Target Analyte List (TAL) metals. One sample was analyzed for metals using the Toxicity Characteristic Leaching Procedure (TCLP) test. All three of the chat samples collected from the Catherine sub site contained lead concentrations that exceeded the EPA Region 7 preliminary remediation goal (PRG) of 400 mg/kg for residential properties. The highest concentration was 2,100 mg/kg. The chat samples also contained moderate to elevated concentrations of other metals including arsenic, cobalt, copper, iron, nickel, and zinc. Results of the TCLP test indicated that the chat has the characteristic of metals toxicity for lead.

Shallow groundwater collected from the chat area contained low to moderate concentrations of cobalt, iron, lead, manganese, nickel, and zinc. Groundwater concentrations for arsenic, iron, lead, manganese, and sulfate exceeded the EPA maximum contaminant levels (MCLs). In addition, the groundwater concentrations exceeded applicable surface water quality criteria for nickel, chloride, and sulfate.

Surface soil samples were collected from five locations surrounding the Catherine site and analyzed for TAL metals. None of the 9 soil samples exceeded the EPA Region 7 PRG for lead.

In general, water quality standards (WQSs) were met for all of the contaminants of potential concern in the surface water of the Catherine pond. The sediment sample in Logtown Branch slightly exceeded the ecological probable effects concentration for lead.

### **1.3.2 Skaggs Sub Site**

A RI report for OU5 was completed during 2008 (BVSPC, 2008). Based on the limited amount of borings installed at the Skaggs sub site, it is estimated that there is approximately 5,000 cubic yards of chat in the western chat pile, 56,500 cubic yards of chat in the central chat pile, and 580 cubic yards of chat in the eastern chat pile. The level of certainty in the estimated volume of chat is low and additional investigations are necessary to collect information that can be used to provide a more reliable estimate of the volume of chat at the site.

During the RI, continuous 5-foot core samples through the chat were collected at 2 locations using a track-mounted geoprobe rig. In addition, core samples through the chat in the eastern and western piles were collected using a hand auger. The chat samples were analyzed for TAL metals. One sample was analyzed for metals using the TCLP test. All 6 of the samples collected from the chat contained lead concentrations that exceeded the EPA Region 7 PRG for residential soils. The highest concentration was 28,400 mg/kg in the sample from the east pile. The chat samples also contained moderate to elevated concentrations of other metals including arsenic, cobalt, copper, iron, nickel, and zinc. Results of the TCLP test indicated that the chat has the characteristic of metals toxicity for lead.

Shallow groundwater collected from the Skaggs area contained low to moderate concentrations of cobalt, iron, lead, manganese, nickel, and zinc. Groundwater concentrations for arsenic, iron, lead, and manganese exceeded EPA MCLs. In addition, the groundwater concentrations exceeded applicable surface water quality criteria for iron, lead, nickel, and zinc.

Surface and subsurface soil samples were collected from five locations surrounding the Skaggs site. Lead concentrations in 5 of the 10 surface and subsurface soil samples exceeded the EPA Region 7 PRG for residential soils.

Four surface soil samples were collected from the floodplain of the unnamed tributary stream that drains south-southwest from the Skaggs area to Plum Creek and one surface soil sample was collected from the floodplain of an unnamed tributary that drains eastward from the Skaggs area to the LSFR. The samples were collected from 15 to 30 ft from the channel and analyzed for TAL metals. Two of the 5 floodplain soil samples exceeded the residential screening levels for lead.

Surface water and sediment samples were collected in an unnamed tributary that drains south-southwest from the Skaggs Mine area toward Plum Creek and one sample was collected from a stock pond immediately southwest of the sub site. One measurable sample for dissolved lead in the stock pond exceeded the WQS for aquatic life. Because the analytical reporting limits

for cadmium, copper, lead, and silver were higher than the WQSSs, it is unknown if water quality within the pond or in the unnamed tributary have been adversely impacted by these constituents. No other metals in the surface waters exceeded the WQSSs.

The data from the eleven sediment samples that were collected suggest that arsenic, copper, iron, lead, manganese, and nickel are of concern in the Skaggs area sediments. Lead is of most concern because it exceeded the human health value in 9 of 11 samples and the ecological benchmark value in 10 of 11 samples. Nickel exceeded the ecological benchmark value of 49 mg/kg in 9 of the samples. Arsenic was slightly elevated in sample 56,300 mg/kg near the center of the sub site. In general, the highest concentrations occur in the drainage east of the site.



## 2.0 Investigation Objectives

The overall quality assurance objective for Madison County Mines OU5 RD is to develop and implement procedures for field sampling and reporting that provide technically and legally defensible results to delineate the nature and extent of contamination, evaluate contaminant migration, and support the remedial design process. Investigation-specific data quality objectives were prepared in accordance with EPA's *Guidance for the Data Quality Objectives Process* (QA/G-4) (EPA, 2000). Primary data quality objectives (DQOs) developed for the Madison County Mines OU5 RD are defined below by environmental medium. More details on these DQOs are provided in the accompanying QAPP.

The primary objective of surface and subsurface soil sampling is to:

- Determine the vertical and horizontal extent of contamination in the vicinity of the chat piles. The data will be used to assist in estimating the volume of soil requiring excavation and transport to the chat piles for disposal.

The primary objective of floodplain soil sampling is to:

- Determine the vertical and horizontal extent of contamination in the floodplain along the intermittent streams. The data will be used to assist in estimating the volume of floodplain soil requiring excavation and transport to the chat pile for disposal.

The primary objective of sediment sampling is to:

- Define the quality of various sediments and potential metal flocculants in streams.

The primary objective of surface water sampling is to:

- Determine whether metal concentrations in the intermittent streams exceed federal and state WQS.

The primary objective of groundwater sampling is to:

- Monitor concentrations in the groundwater around the chat piles to ensure that metals in the chat are not migrating from the Site.



### 3.0 Field Sampling Activities

This section presents the field sampling activities for the Site (both Catherine and Skaggs sub sites) that will be conducted in support of the RD. Documentation of these sampling activities, including photographic records, global positioning system (GPS) location information, and field sampling forms, is described in Section 7.0 of this document.

Surface and subsurface soil samples and floodplain soil samples will be collected around the perimeter of the Catherine and Skaggs chat piles and analyzed for lead using an X-ray Fluorescence Spectroscopy (XRF) instrument. The cleanup goal for lead is 400 mg/kg. The results shall provide the following information:

- Aerial extent of mine waste material at each site.
- Aerial extent of contaminated soil at each site.
- Estimation of the vertical extent of contaminated chat material at each sub site.
- Chemical composition of mining waste material.

GPS coordinates will also be obtained to document the approximate location of each mine shaft observed on the property. BVSPC personnel will maintain a safe distance from the mine shafts while obtaining the coordinates.

Site maps showing the preliminary sample locations of the surface and subsurface soil samples, floodplain soil samples, and surface water/sediment samples are presented in Figures 3-1 through 3-10.

Visual observations will also be made to determine the boundaries of the chat at the in-situ sampling locations and GPS coordinates will be obtained at these locations.

Sediment and surface water samples will be collected once along the Logtown branch and two unnamed intermittent streams.

Eight (8) groundwater monitoring wells will be installed and sampled once for metals to determine if these contaminants are migrating from the Site.

Except to the extent discussed in this section of the FSP, sampling protocols will follow the EPA Region 7 standard operating procedures (SOPs) provided in Appendix A. The following exposure thresholds for each contaminate metal of concern are:

- Lead - 400 mg/kg
- Arsenic – 180 mg/kg
- Cobalt – 130 mg/kg
- Manganese – 2200 mg/kg

- Nickel – 380 mg/kg

Prior to entering private property to collect samples, an access agreement must be signed by the property owner. To the extent possible, access agreements will be signed before the field activities begin. The sampling team will have a copy of the signed access agreement with them during the sampling activities.

## **3.1 Soil Samples**

### **3.1.1 Surface Soils**

Surface soil samples will be collected from the Catherine and Skaggs sub sites to determine the horizontal extent of lead contamination from the chat. The sample locations are shown on Figures 3-1 and 3-2 and listed in Table 3-1. An XRF instrument will be used to obtain in-situ readings to determine the locations where the lead concentration in the soil is less than 400 mg/kg. The final locations of the in-situ samples will be selected using professional judgment to provide representative sampling locations around the chat areas. At each of the in-situ sampling locations, the sampler, based on visual observations, will select an initial sample location that is approximately 15 feet beyond the visual outer edge of the chat. The sampler will obtain the second in-situ sample at a location that is approximately 15 feet beyond the initial location if the lead concentration is greater than 400 mg/kg. Depending upon the sampling results from these two samples, additional in-situ sample readings may have to be obtained in the vicinity of the first two readings to determine the approximate location where the lead concentration is less than 400 mg/kg.

Confirmation surface soil samples will be collected at the rate of 10 percent of the in-situ XRF samples and will be submitted to the EPA Region 7 laboratory for analysis of Resource Conservation and Recovery Act (RCRA) metals (arsenic, barium, cadmium, chromium, lead, selenium, and silver), excluding mercury. All submitted surface samples will also be analyzed for cobalt, manganese, and nickel.

The samples will be analyzed for the constituents listed in Table 3-2; QA/QC sample requirements are shown in Table 3-3. The analytical protocols and methods are specified in Tables B-2 and B-3 of the QAPP. Sample container requirements, preservatives, and holding times are provided in Table B-2 of the QAPP. The relative percent difference between the data from the XRF analyses and the analytical laboratory results should be less than 50 percent.

All of the XRF readings will be recorded in the field logbook. GPS coordinates will be obtained at each sampling point.

### **3.1.2 Subsurface Soils**

Subsurface soil samples will be collected on the Catherine and Skaggs sub sites to determine the horizontal and vertical extent of the lead contamination. In-situ XRF readings will be taken of the surface soil at each location. If the lead concentration is above 400 mg/kg, a test pit will be excavated at that location. A backhoe will be used to excavate the soil at each test pit location. Grab samples will be 1-foot depth intervals and collected from the bucket of the backhoe. It is anticipated that two locations per acre will be sampled. The sample locations are shown on Figures 3-3 and 3-4 and are listed in Table 3-1. The locations of these test pits may be adjusted in the field based on site conditions, the presence of mining and processing related features, and the best professional judgment of the field team leader.

At each location, soil samples will be collected at a vertical interval of 1 foot below ground surface or to a depth of bedrock refusal. XRF readings will be taken of the soil at each 1 foot interval to determine the location where the lead concentration is below 400 mg/kg. At the interval in the test pit where the lead concentration is less than 400 mg/kg, a confirmation sample will be collected. Confirmation soil samples will be collected from 10 percent of the test pits and submitted to the EPA laboratory for analysis of RCRA metals (excluding mercury), cobalt, manganese, and nickel. The estimated depth of the tailings will be recorded on the field sheet at each sampling location.

As indicated in Table A-1 of the QAPP, the relative percent difference between the data from the XRF analyses and the analytical laboratory results should be less than 50 percent.

The samples will be analyzed for the constituents listed in Table 3-2; QA/QC sample requirements are shown in Table 3-3. The analytical protocols and methods are specified in Tables B-2 and B-3 of the QAPP. Sample container requirements, preservatives, and holding times are provided in Table B-2 of the QAPP.

XRF readings will be recorded in the field logbook. GPS coordinates will be obtained at each sampling point.

### **3.1.3 Floodplain Soils**

Floodplain soil sampling will target areas along approximately 11,000 feet of Logtown branch and two unnamed intermittent streams above the bank of the streams. In-situ XRF readings will be taken every 250 feet along each side of the streams to define the horizontal extent of contamination. The locations will vary depending on the topography of the floodplain. At each sampling location, in-situ XRF readings of the surface soil on each side of the stream will be taken to establish the distance from the stream where the lead concentration is below 400 mg/kg. The vertical depth of contamination will be determined by excavating the soil with a shovel at the midpoint between the stream bed and the location where the lead concentration in

the surface soil is less than 400 mg/kg. In-situ XRF readings will be taken in the excavated area at six inch intervals until the depth where the lead concentration is below 400 mg/kg is reached and the depth will be recorded. Locations of the floodplain soil samples are shown in Figures 3-5, 3-6, and 3-7 and are listed in Table 3-1.

The samples will be analyzed for the constituents listed in Table 3-2; XRF readings will be recorded in the field logbook. GPS coordinates will be obtained at each sampling point.

## **3.2 Water/Sediment Samples**

### **3.2.1 Surface Water**

Surface water samples will be collected from six (6) locations along the Logtown branch and two unnamed intermittent streams. Filtered and unfiltered samples will be collected at each location and submitted to the EPA laboratory for analysis of total and dissolved RCRA metals (excluding mercury) and miscellaneous water quality parameters, including chloride, sulfate acidity, conductivity, total and suspended solids, alkalinity, and hardness. The samples will also be tested for nickel. The sampling methods and protocols will follow the EPA Region 7 SOP No. 4334.8A presented in Appendix A. Surface water sampling locations are shown in Figures 3-8, 3-9, and 3-10 and are listed in Table 3-1. The samples will be analyzed for the constituents listed in Table 3-2; QA/QC sample requirements are shown in Table 3-3. The analytical protocols and methods are specified in Tables B-2 and B-3 of the QAPP. Sample container requirements, preservatives, and holding times are provided in Table B-2 of the QAPP.

GPS coordinates will be obtained at each sampling point.

### **3.2.2 Sediment**

Sediment samples will be collected from the same six (6) locations along the Logtown branch and two unnamed intermittent streams (Figures 3-8, 3-9, and 3-10). All surface water samples will be collected prior to the sediment collection. If it is necessary to wade into the water, the team member will be careful not to disturb bottom sediments at the sample collection points. Subsequently, the team member should approach the sample collection from downstream. Depending on the character and accessibility of the sediments, a shovel, spoon, dredge, or hand auger will be used to collect the samples. With a clean stainless steel bowl and sampling equipment immediately available and while wearing uncontaminated gloves the sediment sample will be collected while facing upstream and placed into the sample container. All of the sediment samples will be submitted to the EPA Region 7 laboratory for analysis of nickel and RCRA metals, excluding mercury.

The samples will be analyzed for the constituents listed in Table 3-2; QA/QC sample requirements are shown in Table 3-3. The analytical protocols and methods are specified in

Tables B-2 and B-3 of the QAPP. Sample container requirements, preservatives, and holding times are provided in Table B-2 of the QAPP.

### **3.2.3 Groundwater Wells**

Eight (8) groundwater monitoring wells will be installed in the Catherine and Skaggs sub sites around the proposed repositories. Two of the wells (one on each sub site) will be designated as a background well. The locations of the groundwater monitoring wells will be determined following the design of the cap over the Catherine and Skaggs repositories.

It is assumed that each of the wells will be approximately 20 feet deep. The wells will be developed and sampled using a peristaltic pump and high capacity 0.45 µm certified filter. Filtered and unfiltered samples will be collected and the samples will be submitted to the EPA Region 7 laboratory for analysis of RCRA metals, excluding mercury; manganese; and water quality parameters. Sampling methods and protocols will follow EPA SOP 4334.15A presented in Appendix A.

The sampling locations will be determined after the repository locations have been completed. The samples will be analyzed for the constituents listed in Table 3-2; QA/QC sample requirements are shown in Table 3-3. The analytical protocols and methods are specified in Tables B-2 and B-3 of the QAPP. Sample container requirements, preservatives, and holding times are provided in Table B-2 of the QAPP.

Static water level measurements will be taken measured from the top of the well casing. A surveyor will be procured to survey the top of casing elevations. GPS coordinates will be obtained at each well location.

## **3.3 Sample Handling**

Sample Hand Sample packaging and shipping procedures are based on EPA guidance and U.S. Department of Transportation (USDOT) regulations (49 CFR). Samples collected at the Madison County Mines OU5 Site will be shipped as non-dangerous goods.

The following general steps will be used for the packaging and shipping of samples:

- 1) Affix a completed sample label to the sample container and protect it by covering with clear tape.
- 2) Protect each glass sample container with bubble wrap or foam, taped securely in place as necessary.
- 3) Tape the shipping container drain closed.
- 4) Place packing material in bottom of cooler for cushioning as necessary.

- 5) Place the samples (protected by bubble wrap or foam) into the cooler.
- 6) Fill the remaining volume of the cooler with packing material as necessary.
- 7) Fill out and sign the chain-of-custody record and indicate the estimated time the shipping container will be relinquished to a courier service or when the shipping container will be relinquished directly to the laboratory.
- 8) Make a copy of the form for the project file and seal the original form and copies in a sealable plastic bag and tape it to the inside lid of the shipping container.
- 9) Secure shipping container by making several revolutions with strapping tape or clear plastic tape on both ends.
- 10) If shipped by courier, place airbill marked for delivery with laboratory address on shipping container.
- 11) Affix custody seals over top front and top back corners of the shipping container. Cover seals with clear plastic tape.
- 12) Notify the EPA laboratory by telephone and provide the following information:
  - Sender's name and firm (BVSPC).
  - Project name (Madison County Mines OU5 Site).
  - Number and type of samples to be received.
  - Date and estimated time of delivery.
  - Anticipated sampling schedule (for establishing sample delivery groups at laboratory).

The laboratory that will perform the analyses is listed below:

EPA Region 7 Laboratory  
c/o Nicole Roblez  
300 Minnesota Avenue  
Kansas City, KS 66101  
(913) 551-5130

- 13) The BVSPC Site Manager will maintain a file containing copies of the documentation.



## 4.0 Sample Numbering System

A sample numbering system will be used to uniquely identify each sample that is collected and analyzed as part of this project. Samples will be given two identification numbers: 1) a unique BVSPC sample identifier and 2) a tracking code for samples submitted to the EPA Region 7 laboratory. These numbers will be cross-referenced in field notebooks, on sample collection data sheets, and in the project database.

The BVSPC sample number will consist of six alphabetic characters followed by a station identification number which is unique to each sample location. The alphabetic characters will provide information about each sample in order as follows:

1. Residential sample location
  - A Front yard sample, the left quadrant (when facing the front of the house) - F1
  - B Front yard sample, the right quadrant (when facing the front of the house) - F2
  - C Back yard sample, directly behind F1 - B1
  - D Back yard sample, directly behind F2 - B2
  - E Drip zone sample
  - G Garden area sample
  - H Home lobby or entryway
  - I Living Area location
  - J Bedroom location
  - P Play area sample
  - R Road/Driveway sample
  - S Chat Pile
  - T Chat Pile
  - U Chat Pile
  - V Chat Pile
  - Z Residential location not applicable
2. Sample matrix
  - A Surface water
  - B Sediment
  - G Groundwater
  - P Potable water
  - S Soil
  - Z Sample matrix not applicable

3. Sample derivation
  - C Composite
  - G Grab/Discrete
  - Z Sample derivation not applicable
4. Sample quality control status
  - P Primary environmental sample
  - D Field duplicate
  - E Equipment rinsate
  - M Matrix spike/matrix spike duplicate
  - Z Sample quality control status not applicable
5. Analytical method
  - X Analyzed by field XRF
  - L Analyzed by offsite laboratory
  - Z Analytical method not applicable
6. Sample interval
  - A 0 - 1 inch
  - B 0 - 6 inches
  - C 0 - 12 inches
  - D 1 - 2 feet
  - E 2 - 3 feet
  - F 3 - 4 feet
  - G 4 - 5 feet
  - H 5 - 6 feet
  - I 6 - 7 feet
  - J 7 - 8 feet
  - K 8 - 9 feet
  - L 9 - 10 feet
  - M 10 - 11 feet
  - N 11 - 12 feet
  - O 12 - 13 feet
  - P 13 - 14 feet
  - Q 14 - 15 feet
  - R 15 - 16 feet
  - S Greater than 16 feet
  - Z Sampled interval not applicable

The station number uniquely identifies sample locations and will be assigned as indicated below. Each sampled location will require a unique five digit code.

#### Catherine Sub site

- Surface soils 28200 to 28399
- Subsurface soils (test pits) 1 to 99
- Floodplain soils 52301 to 52399
- Surface water/Sediment 52400 to 52410
- Groundwater wells 28011 to 28014

#### Skaggs Sub site

- Surface soils 30200 to 30399
- Subsurface soils (test pits) 100 to 199
- Floodplain soils east of Skaggs 56301 to 56399
- Surface water/Sediment east of Skaggs 56400 to 56010
- Floodplain soils south of Skaggs 57701 to 57899
- Surface water/Sediment south of Skaggs 57900 to 57910
- Groundwater wells 30011 to 30014

Based upon the preceding, a primary surface water sample collected at Station location 56400 east of the Skaggs sub site at OU5 would have the following sample ID:

ZAGPLZ-56400

The samples that are submitted to the EPA laboratory will be assigned a corresponding EPA sample identification number consisting of three components: the activity number (EPA assigned), ASR Number (EPA assigned), the unique sample identification number and the sample qualifier (if applicable). The following is an example of a completely numbered field duplicate, with each component identified:

1195-001-FD

where:

1195 = EPA ASR Number  
001 = Sample Identification Number  
FD = Sample Qualifier (QC Code)

If the sample is a primary sample, rinsate, or MS/MSD sample, no qualifier will be

designated. Rinsate samples will be assigned their own unique identification number and will be noted as a rinsate sample on the field sheet. If required by the EPA Region 7 laboratory, the MS/MSD samples will consist of a triple volume of a primary sample and will be noted as a MS/MSD on the field sheet and chain-of-custody record.

Field duplicate samples will be assigned the same sample identification number as the primary sample, but will be qualified as FD. Trip blank samples will not be required.

The sample collection field sheets and field logbook will also include information concerning the sample matrix, location, and sample depth, if applicable.

## **5.0 Equipment Decontamination Procedures**

Procedures for equipment decontamination will be implemented to avoid cross-contamination of samples of various media that are to be submitted for chemical analysis. Sampling equipment will be thoroughly cleaned and decontaminated before initial use and between sample locations. The types of equipment requiring decontamination may include spoons, bowls, augers, buckets, sieves, and shovels.

Decontamination of the sampling equipment will include the following:

- Wash equipment with dilute Alconox(c) or Liquinox(c) wash.
- Circulate and rinse with potable water.
- Rinse with distilled water.
- Rinse with isopropanol
- Air dry in clean environment
- Wrap in aluminum foil or seal in a plastic bag until the next use.



## **6.0 Investigation-Derived Waste**

Investigation derived waste (IDW) will include soil cuttings and decontamination liquids generated during the installation of the groundwater monitoring wells; decontamination fluids from the soil, surface water, and sediment sampling; soil samples analyzed only by XRF, disposable personal protective equipment (PPE), and trash. All IDW consisting of soil cuttings, excess soil samples, and decontamination liquids will be containerized and disposed of at the EPA repository located at the Catherine chat area.

### **6.1 Liquids**

Liquid IDW will consist of decontamination fluids. Liquids generated during decontamination of the drill rig and sampling equipment will be containerized and disposed of at the EPA repository at the Catherine site.

### **6.2 Personal Protective Equipment**

Disposable PPE and trash will be bagged in plastic bags and will be disposed of as municipal solid waste.

### **6.3 Solids**

The unused portions of soil, mine wastes, sediment, and other solids will be separated and disposed of at the EPA repository at the Catherine site.





## **7.0 Documentation**

### **7.1 Sample Station Information**

At the time of sampling or when sample stations are located, stations will be flagged or otherwise clearly marked. Sample station information will be recorded in field log books or downloaded directly from GPS instruments. In both cases, station information will be maintained in the project database.

#### **7.1.1 Location Information**

The location of each sampling station will be determined using GPS technology and the location will be marked on base maps or aerial photos. Coordinates will be recorded in state plane coordinates (Missouri Eastern Zone) recorded to four decimals. Instruments will be downloaded as appropriate and the data entered into the project data base.

Unique features at sample station locations will be visually described and this information recorded in field notebooks.

#### **7.1.2 Photographs**

It is not anticipated that all sampling locations will be photographed. If any sampling locations are photographed, they will be photographed in a manner to permit recognition of the location as much as possible. Digital images will be downloaded and stored as digital files daily. An entry will be made in a field notebook to identify which sampling station is depicted in each photograph. The photographs will show the surrounding area and reference objects that identify the sampling sites. Logbook entries of photographs will have four major components: photographer's initials, frame number, date, and a description of what was photographed. For example: GLF, 1-1, 09-24-13, Sampling at surface water location 25,000.

### **7.2 Sample Collection Information**

The most important aspect of sample custody and documentation is thorough, accurate recordkeeping. Sample collection information will be recorded on field sample data sheets and field parameter measurement sheets, supplemented with field notebooks as necessary.

Information will be recorded using permanent, waterproof ink. Field notebooks will be copied periodically during sample collection efforts (to the extent practical) or upon completion of the field sampling task.

### **7.2.1 Sample Data Sheets**

Field sample data sheets will be used to track access to properties, the attributes of each sample, and the laboratory analyses to be performed. Example field sample sheets for samples that are submitted to the laboratory and samples that are analyzed only with an XRF instrument are provided in Appendix B. The field team will complete a field sheet for each sample at the time of collection. The field sheet includes the sample location, BVSPC sample identification number, EPA sample identification number, sample description, analyses to be run, and field sketches and other descriptive information.

Field parameter sample sheets will also be prepared for all water samples in which field parameters are measured at the time of sample collection. An example of this field data sheet is shown in Appendix B. The sheet includes sample identification and station location and field parameter measurements for the sample.

### **7.2.2 Field Notebooks**

All information obtained during the sampling activities will be recorded in bound notebooks with consecutively numbered pages. All entries in notebooks and on sample documentation forms will be made in waterproof ink, and corrections will consist of line-out deletions that are initialed and dated. Entries in the logbook may include the following, as applicable:

- Name and title of author, date and time of entry, and physical/environmental conditions during field activity.
- Purpose of sampling activity.
- Name and address of field contact.
- Names and titles of field crew members.
- Names, titles, and affiliations of any site visitors.
- Type of waste, suspected waste concentration if known, and sample matrix.
- Sample collection method.

- Number and volume of samples taken.
- Location, description, and log of photographs of the sampling activities and locations.
- References for all maps and photographs of the sampling site(s).
- Information concerning sampling changes, scheduling modifications, and change orders
- Details of the sampling location.
- Date and time of collection.
- Field observations.
- Any field measurements made (such as pH, specific conductivity, temperature).
- Sample identification numbers.
- Information from container labels of reagents used, HPLC water used for blanks, etc.
- Sample preservation.
- Sample distribution and transportation (such as name of the laboratory and approved carrier).
- All sample documentation, such as the following:
  - Bottle QC lot numbers as received from repository.
  - Activity numbers as received from the EPA.
  - Chain-of-custody record numbers as received from the EPA.
- Decontamination procedures.
- All documentation concerning derived wastes, such as the following:
  - Contents and approximate volume of waste in each drum.
  - Type and predicted level of contamination.
- Summary of daily tasks (including costs) and scope of work changes required by field conditions.
- Signature of the personnel responsible for observations and the date.

Sampling situations vary widely. No general rules can specify the exact information that must be entered in a notebook for a particular site. However, the notebook must contain sufficient information so that someone can reconstruct the sampling activity without relying on the collector's memory. The notebooks must be kept in the field team member's possession or in a secure place during the investigation. Following the investigation, the notebooks will become

a part of the final project file.

## **7.3 Sample Tracking Information**

The following subsections describe the required sample tracking documentation that will be used for the Madison County Mines OU 5 investigation. The documents will be used for each environmental sample collected for off-site laboratory analysis.

### **7.3.1 Container Labels**

An adhesive sample label will be placed on each sample container submitted for chemical analysis. The following information will be included on each sample label:

- Site name.
- EPA sample identification number.
- Name of sampler.
- Sample collection date and time.
- Analysis requested and preservatives added.

Information known before field activities (i.e., site name, sample numbers, etc.) may be pre-printed on the sample labels. Duplicate sample labels will be prepared for cases when various aliquots of a sample must be submitted separately for individual analyses.

### **7.3.2 Chain-of-Custody Forms and Custody Seals (COC)**

A COC record will be completed for each sample shipment. Standard EPA Region 7 laboratory COC records will be used. After completion of the COC record, a photocopy of the COC will be made and both copies of the original COC record will be enclosed in a sealable plastic bag and secured to the inside of the shipping container lid. The photocopy will be retained for reference before receipt of the original forms with the laboratory's data deliverable package. The original COC record received from the laboratory will be placed in the final project file.

Custody seals will be used to ensure the integrity of the samples should they remain unattended or when they are relinquished to a delivery service until they are opened by the

laboratory. All samples will be shipped in an insulated shipping container, and each shipping container will be sealed with at least two signed custody seals. The seals will be affixed to each shipping container so that it is necessary to break the seals to open the shipping container.

As long as the COC record is sealed inside the shipping container and the custody seals remain intact, commercial carriers will not be required to sign the COC record.

### **7.3.3    *Airbills and Bills of Lading***

An airbill or bill of lading will be completed for each cooler shipment. Next morning overnight delivery will be indicated on the airbill by the field personnel shipping the samples. More than one shipping container may be forwarded to the same address under one airbill. When the shipping containers are relinquished to the delivery service, additional information will be added to the airbill by an employee of the delivery service, and a copy of the airbill will be received by the field personnel.

### **7.3.4    *Sample Receipt Forms***

If split samples are provided to another party, a receipt for samples form will be completed. After completion of this form, the original copy, which is to be signed, will be retained for the project file, and a copy will be given to the sample recipient.



## **8.0 Field Activities Schedule**

It is anticipated that the sampling activities specified in this FSP will begin in late 2013 or early 2014 and if weather permits will last for approximately four (4) weeks.





## 9.0 Bibliography

- Anonymous. *Madison County Expected to Become Great Mining Center*. Article from unknown newspaper, date unknown.
- Black & Veatch Special Projects Corp., 2004. *Remedial Investigation/Feasibility Study Field Sampling Plan for Soil Sampling in Residential Yards*. Madison County Mines OU3 Site, Fredericktown, Missouri. Prepared for USEPA Region VII.
- Black & Veatch Special Projects Corp., 2008. *Madison County Mines Site, Remedial Investigation Report*. Madison County Missouri. April 2008.
- Black & Veatch Special Projects Corp., 2011. *Remedial Investigation/Feasibility Study Field Sampling Plan for Soil Sampling in Residential Yards*. Madison County Mines OU4 Site, Fredericktown, Missouri. Prepared for USEPA Region VII.
- Black & Veatch Special Projects Corp., 2012. *Remedial Investigation/Feasibility Study Field Sampling Plan for Soil Sampling in Residential Yards*. Madison County Mines OU5 Site, Fredericktown, Missouri. Prepared for USEPA Region VII.
- Black & Veatch Special Projects Corp., 2013. *Quality Assurance Project Plan, Field Investigation for Remedial Design*. Madison County Mines Operable Unit 5 Site, Madison County, Missouri.
- Boone, M., 2001. *St. Francis River Watershed Inventory and Assessment*. Missouri Department of Conservation.
- Bouchard, W.L., date unknown. *Inspection Trip of Historic Mine La Motte Mines*. Partial copy of article from the Lead Belt News.
- Burt, D.M., 1981. *Acidity-Salinity Diagrams, Application to Greisen and Porphyry Deposits*. *Economic Geology*, vol. 76, pp. 832-843.
- Dames & Moore, 1990 (April 3). *Madison Mine, Preliminary Site Characterization Report*.

Democrat News, 1947 (February 20). *Hickory Nut Tract Being Drilled Now*. Newspaper article.

Grawe, Oliver R., date unknown. *Pyrites Deposits of Missouri*. Missouri Geological Survey and Water Resources.

Hall, G.S., 1939. *History of Mine La Motte*.

James, Jack A., 1949. *Geologic Relationships of the Ore Deposits in the Fredericktown Area, Missouri*. State of Missouri, Division of Geological Survey and Water Resources, Report of Investigations No. 8, 22 pp. plus plates.

Madison County, 2002. 2002 Plat Book.

Missouri Bureau of Mines and Mine Inspection (MBMMI), 1904. *Lead, Zinc, Coal and Other Mines, Year Ending December 31, 1904*. 18th Annual Report of the Bureau of Mines and Mine Inspection of the State of Missouri.

Payne, R., 1893 (March 23). *Einstein Silver Mines*. Letter to C.F. Marbut, Esq. reprinted in A Geological and Mining History of Silver Mines Recreation Area, Mark Twain National Forest District Ranger.

Skaggs, J.P. 2004. Personal communication (conversation) with Jack Mozingo, October 22, 2004.

Shoemaker, F.C., date unknown. *Madison County: Land of Mines, Forests, Farms, and Factories*. In: Missouri Historical Review.

Tetra Tech 2003. *Removal Assessment Summary Report, Madison Mine Site, Fredericktown, MO*. February 2003.

Tolman, Carl, 1933. *The Geology of the Silver Mine Area, Madison County, Missouri*. Missouri Bureau of Geology and Mines, Reprint of Appendix I, 57th Biennial Report, 39 pp.

- U.S. Census Bureau. 2001. Census 2000 Summary File 1. Produced by Missouri Census Data Center.  
[http://mcdc2.missouri.edu/webrepts/sdcprofiles1/mo/160\\_29-25768\\_Fredericktown\\_city](http://mcdc2.missouri.edu/webrepts/sdcprofiles1/mo/160_29-25768_Fredericktown_city)
- U.S. Environmental Protection Agency (EPA), 1988 (October). *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, Interim Final*, EPA/540/G-89/004.
- EPA, 1992 (July). *Indoor Radon and Radon Decay Product Measurement Device Protocols*, Office of Radiation Programs, EPA 402/R-92-004.
- EPA, 2000. *Guidance for the Data Quality Objectives Process*, EPA Office of Research and Development, EPA QA/G-4.
- EPA, 2003 (September). *Aerial Photographic Analysis of Madison Mines Study Area, Fredericktown, Missouri, EPA Region 7*, EPA Environmental Services Division, Report TS-PIC-20307003S.
- EPA, 2005 (April). Quality Assurance Project Plan (QAPP) for Sampling Heavy Metals, Madison Mines Site, Fredericktown, Missouri, CERCLIS ID No. MODO 98633415-OU3.
- U.S. Geological Survey, Missouri Division of Geological Survey and Water Resources, and Army Corps of Engineers (USGS et al.), 1967 (April 6). *Mineral and Water Resources of Missouri*. Mineral and Water Resources of Missouri, vol. XLIII, Second Series, Missouri Department of Business Administration.



## TABLES



Table 3-1  
Sampling Locations  
Remedial Design  
Madison County Mines OU5  
Madison County, Missouri

Sample Location	Sample Number	Media	Northing	Easting	Comments
<b>Surface Soil Samples</b>					
SS-1	28200	Surface Soil	641414.9379	866624.4567	Catherine
SS-2	28201	Surface Soil	641375.9448	866699.5722	Catherine
SS-3	28202	Surface Soil	641328.2915	866774.6645	Catherine
SS-4	28203	Surface Soil	641286.2380	866850.9412	Catherine
SS-5	28204	Surface Soil	641218.2557	866917.9747	Catherine
SS-6	28205	Surface Soil	641146.8907	866987.1966	Catherine
SS-7	28206	Surface Soil	641074.1529	867056.8960	Catherine
SS-8	28207	Surface Soil	641003.6975	867127.2039	Catherine
SS-9	28208	Surface Soil	640936.3757	867190.6234	Catherine
SS-10	28209	Surface Soil	640866.3448	867243.2284	Catherine
SS-11	28210	Surface Soil	640769.2109	867228.7142	Catherine
SS-12	28211	Surface Soil	640698.1655	867299.0668	Catherine
SS-13	28212	Surface Soil	640618.7997	867359.2490	Catherine
SS-14	28213	Surface Soil	640532.1710	867414.3579	Catherine
SS-15	28214	Surface Soil	640451.4293	867474.6204	Catherine
SS-16	28215	Surface Soil	640363.4564	867524.6296	Catherine
SS-17	28216	Surface Soil	640274.7365	867576.2331	Catherine
SS-18	28217	Surface Soil	640167.0365	867582.6536	Catherine
SS-19	28218	Surface Soil	640168.2321	867477.7969	Catherine
SS-20	28219	Surface Soil	640181.6014	867376.8691	Catherine
SS-21	28220	Surface Soil	640234.3792	867294.9247	Catherine
SS-22	28221	Surface Soil	640287.1569	867215.0636	Catherine
SS-23	28222	Surface Soil	640283.6847	867156.7302	Catherine
SS-24	28223	Surface Soil	640216.3236	867081.0358	Catherine
SS-25	28224	Surface Soil	640180.2125	866990.0636	Catherine
SS-26	28225	Surface Soil	640178.1292	866890.0636	Catherine
SS-27	28226	Surface Soil	640212.1569	866799.7858	Catherine
SS-28	28227	Surface Soil	640237.3224	866678.4304	Catherine
SS-29	28228	Surface Soil	640279.3722	866583.6577	Catherine
SS-30	28229	Surface Soil	640296.2962	866485.1002	Catherine
SS-31	28230	Surface Soil	640326.4278	866389.8839	Catherine
SS-32	28231	Surface Soil	640364.0750	866300.5005	Catherine
SS-33	28232	Surface Soil	640453.6482	866256.2682	Catherine
SS-34	28233	Surface Soil	640510.1516	866176.5604	Catherine
SS-35	28234	Surface Soil	640585.9017	866152.8728	Catherine
SS-36	28235	Surface Soil	640673.1922	866123.6798	Catherine
SS-37	28236	Surface Soil	640731.2921	866096.9213	Catherine
SS-38	28237	Surface Soil	640762.8324	866227.8302	Catherine
SS-39	28238	Surface Soil	640855.7759	866191.2631	Catherine
SS-40	28239	Surface Soil	640936.9563	866136.6455	Catherine
SS-41	28240	Surface Soil	641028.6130	866141.1287	Catherine
SS-42	28241	Surface Soil	641083.2812	866214.3520	Catherine
SS-43	28242	Surface Soil	641153.3555	866260.0587	Catherine
SS-44	28243	Surface Soil	641192.4374	866338.5140	Catherine
SS-45	28244	Surface Soil	641211.8368	866431.8808	Catherine
SS-46	28245	Surface Soil	641252.7154	866518.8510	Catherine
SS-47	28246	Surface Soil	641298.3051	866564.8273	Catherine
SS-48	28247	Surface Soil	641349.6509	866625.0053	Catherine
SS-49	30200	Surface Soil	639642.1876	868049.8895	Skaggs

Table 3-1  
Sampling Locations  
Remedial Design  
Madison County Mines OU5  
Madison County, Missouri

Sample Location	Sample Number	Media	Northing	Easting	Comments
SS-50	30201	Surface Soil	639573.4376	868126.9728	Skaggs
SS-51	30202	Surface Soil	639506.7709	868195.0284	Skaggs
SS-52	30203	Surface Soil	639441.4931	868268.6395	Skaggs
SS-53	30204	Surface Soil	639380.3820	868347.1117	Skaggs
SS-54	30205	Surface Soil	639309.5487	868421.4172	Skaggs
SS-55	30206	Surface Soil	639238.7153	868492.2506	Skaggs
SS-56	30207	Surface Soil	639159.5487	868555.4450	Skaggs
SS-57	30208	Surface Soil	639072.7431	868507.5284	Skaggs
SS-58	30209	Surface Soil	638976.9098	868481.8339	Skaggs
SS-59	30210	Surface Soil	638889.1179	868539.0299	Skaggs
SS-60	30211	Surface Soil	638797.3405	868583.1120	Skaggs
SS-61	30212	Surface Soil	638701.2271	868604.7917	Skaggs
SS-62	30213	Surface Soil	638604.3910	868575.1627	Skaggs
SS-63	30214	Surface Soil	638529.9574	868509.4010	Skaggs
SS-64	30215	Surface Soil	638529.9574	868410.3970	Skaggs
SS-65	30216	Surface Soil	638441.7932	868363.4243	Skaggs
SS-66	30217	Surface Soil	638413.6096	868272.3695	Skaggs
SS-67	30218	Surface Soil	638389.7619	868177.7014	Skaggs
SS-68	30219	Surface Soil	638439.6253	868096.0412	Skaggs
SS-69	30220	Surface Soil	638430.9534	867994.1465	Skaggs
SS-70	30221	Surface Soil	638385.4260	867913.9316	Skaggs
SS-71	30222	Surface Soil	638339.1759	867843.8339	Skaggs
SS-72	30223	Surface Soil	638259.6837	867793.9706	Skaggs
SS-73	30224	Surface Soil	638175.8555	867728.2088	Skaggs
SS-74	30225	Surface Soil	638081.1874	867700.0251	Skaggs
SS-75	30226	Surface Soil	637982.9061	867679.0681	Skaggs
SS-76	30227	Surface Soil	637893.2966	867631.3727	Skaggs
SS-77	30228	Surface Soil	637796.4606	867616.9196	Skaggs
SS-78	30229	Surface Soil	637727.8081	867554.0484	Skaggs
SS-79	30230	Surface Soil	637765.3863	867467.3296	Skaggs
SS-80	30231	Surface Soil	637745.8746	867372.6615	Skaggs
SS-81	30232	Surface Soil	637722.0269	867274.3802	Skaggs
SS-82	30233	Surface Soil	637745.8746	867178.9895	Skaggs
SS-83	30234	Surface Soil	637804.4098	867257.7591	Skaggs
SS-84	30235	Surface Soil	637854.2731	867345.2006	Skaggs
SS-85	30236	Surface Soil	637914.9763	867419.6342	Skaggs
SS-86	30237	Surface Soil	637996.6365	867472.3882	Skaggs
SS-87	30238	Surface Soil	638071.0702	867529.4781	Skaggs
SS-88	30239	Surface Soil	638161.4023	867567.0563	Skaggs
SS-89	30240	Surface Soil	638245.2305	867616.1969	Skaggs
SS-90	30241	Surface Soil	638332.6720	867663.8923	Skaggs
SS-91	30242	Surface Soil	638412.1643	867723.1502	Skaggs
SS-92	30243	Surface Soil	638506.8324	867709.4197	Skaggs
SS-93	30244	Surface Soil	638581.9887	867646.5485	Skaggs
SS-94	30245	Surface Soil	638675.9341	867622.7009	Skaggs
SS-95	30246	Surface Soil	638773.4928	867606.0797	Skaggs
SS-96	30247	Surface Soil	638871.7741	867618.3649	Skaggs
SS-97	30248	Surface Soil	638959.2156	867663.1696	Skaggs
SS-98	30249	Surface Soil	639026.4227	867737.6033	Skaggs
SS-99	30250	Surface Soil	639074.8407	867834.4393	Skaggs



Table 3-1  
Sampling Locations  
Remedial Design  
Madison County Mines OU5  
Madison County, Missouri

Sample Location	Sample Number	Media	Northing	Easting	Comments
SS-100	30251	Surface Soil	639163.0049	867877.7988	Skaggs
SS-101	30252	Surface Soil	639256.9503	867853.9511	Skaggs
SS-102	30253	Surface Soil	639354.5090	867832.9940	Skaggs
SS-103	30254	Surface Soil	639435.4466	867881.4121	Skaggs
SS-104	30255	Surface Soil	639506.2670	867949.3418	Skaggs
SS-105	30256	Surface Soil	639605.9936	867951.5098	Skaggs
<b>Surface/Subsurface Soil Samples</b>					
TP-1	1	Subsurface	641349.5461	866640.5603	Catherine
TP-2	2	Subsurface	641247.3078	866687.2978	Catherine
TP-3	3	Subsurface	641254.9204	866840.1086	Catherine
TP-4	4	Subsurface	641026.1347	866239.2590	Catherine
TP-5	5	Subsurface	641113.7188	866324.8966	Catherine
TP-6	6	Subsurface	641157.5107	866502.9838	Catherine
TP-7	7	Subsurface	641059.2219	866442.6483	Catherine
TP-8	8	Subsurface	641031.9736	866699.5612	Catherine
TP-9	9	Subsurface	641141.9402	866637.2792	Catherine
TP-10	10	Subsurface	641048.5172	866846.5074	Catherine
TP-11	11	Subsurface	641119.5577	866930.1985	Catherine
TP-12	12	Subsurface	641056.6571	867039.8782	Catherine
TP-13	13	Subsurface	640918.2832	866214.4424	Catherine
TP-14	14	Subsurface	640838.0442	866308.8412	Catherine
TP-15	15	Subsurface	640947.7828	866428.0198	Catherine
TP-16	16	Subsurface	640833.3244	866458.6992	Catherine
TP-17	17	Subsurface	640830.9645	866646.3169	Catherine
TP-18	18	Subsurface	640954.8628	866626.2572	Catherine
TP-19	19	Subsurface	640961.9429	866825.6748	Catherine
TP-20	20	Subsurface	640855.7439	866890.5740	Catherine
TP-21	21	Subsurface	640843.9441	867030.9920	Catherine
TP-22	22	Subsurface	640930.0830	867100.6113	Catherine
TP-23	23	Subsurface	640859.4730	867217.9219	Catherine
TP-24	24	Subsurface	640730.6658	866286.4216	Catherine
TP-25	25	Subsurface	640599.6874	866331.2608	Catherine
TP-26	26	Subsurface	640723.5857	866415.0398	Catherine
TP-27	27	Subsurface	640617.3871	866472.8592	Catherine
TP-28	28	Subsurface	640674.0265	866600.2976	Catherine
TP-29	29	Subsurface	640695.2663	866726.5559	Catherine
TP-30	30	Subsurface	640622.1069	866894.1136	Catherine
TP-31	31	Subsurface	640746.0057	866863.4342	Catherine
TP-32	32	Subsurface	640736.5657	867086.4512	Catherine
TP-33	33	Subsurface	640622.1069	867036.8919	Catherine
TP-34	34	Subsurface	640741.2855	867211.5297	Catherine
TP-35	35	Subsurface	640643.3467	867274.0689	Catherine
TP-36	36	Subsurface	640526.5284	866302.9413	Catherine
TP-37	37	Subsurface	640415.6097	866325.3608	Catherine
TP-38	38	Subsurface	640422.6897	866453.9794	Catherine
TP-39	39	Subsurface	640522.9884	866489.3789	Catherine
TP-40	40	Subsurface	640409.7097	866661.6568	Catherine
TP-41	41	Subsurface	640528.8883	866667.5567	Catherine
TP-42	42	Subsurface	640472.2490	866815.0547	Catherine
TP-43	43	Subsurface	640492.3087	866941.3130	Catherine

Table 3-1  
Sampling Locations  
Remedial Design  
Madison County Mines OU5  
Madison County, Missouri

Sample Location	Sample Number	Media	Northing	Easting	Comments
TP-44	44	Subsurface	640533.6081	867077.0116	Catherine
TP-45	45	Subsurface	640421.5096	867131.2907	Catherine
TP-46	46	Subsurface	640532.4283	867349.5881	Catherine
TP-47	47	Subsurface	640429.7694	867265.8091	Catherine
TP-48	48	Subsurface	640510.0084	867422.7471	Catherine
TP-49	49	Subsurface	640402.6297	867453.4268	Catherine
TP-50	50	Subsurface	640345.9907	866396.1599	Catherine
TP-51	51	Subsurface	640311.7710	866516.5186	Catherine
TP-52	52	Subsurface	640317.6709	866688.7965	Catherine
TP-53	53	Subsurface	640252.7717	866744.2557	Catherine
TP-54	54	Subsurface	640335.3706	866929.5132	Catherine
TP-55	55	Subsurface	640231.5319	866877.5940	Catherine
TP-56	56	Subsurface	640335.3706	867126.5709	Catherine
TP-57	57	Subsurface	640243.3318	867068.7514	Catherine
TP-58	58	Subsurface	640312.9511	867259.9092	Catherine
TP-59	59	Subsurface	640215.0123	867361.3880	Catherine
TP-60	60	Subsurface	640315.3110	867514.7860	Catherine
TP-61	61	Subsurface	640204.3922	867480.5662	Catherine
TP-62	100	Subsurface	639572.6783	867858.3335	Skaggs
TP-63	101	Subsurface	639493.0036	867967.0954	Skaggs
TP-64	102	Subsurface	639616.9416	868077.1221	Skaggs
TP-65	103	Subsurface	639506.9150	868093.5630	Skaggs
TP-66	104	Subsurface	639381.7124	867826.7168	Skaggs
TP-67	105	Subsurface	639351.3601	867946.8609	Skaggs
TP-68	106	Subsurface	639296.9790	868144.1499	Skaggs
TP-69	107	Subsurface	639409.5352	868079.6516	Skaggs
TP-70	108	Subsurface	639321.0078	868356.6153	Skaggs
TP-71	109	Subsurface	639401.9470	868242.7947	Skaggs
TP-72	110	Subsurface	639159.1296	867811.5406	Skaggs
TP-73	111	Subsurface	639178.0997	867958.2427	Skaggs
TP-74	112	Subsurface	639089.5726	868058.1520	Skaggs
TP-75	113	Subsurface	639213.5107	868103.6805	Skaggs
TP-76	114	Subsurface	639114.8659	868324.9986	Skaggs
TP-77	115	Subsurface	639231.2160	868314.8811	Skaggs
TP-78	116	Subsurface	639224.8926	868464.1128	Skaggs
TP-79	117	Subsurface	639098.4253	868514.6996	Skaggs
TP-80	118	Subsurface	639018.7506	867747.0421	Skaggs
TP-81	119	Subsurface	638907.4595	867697.7200	Skaggs
TP-82	120	Subsurface	638912.5182	867879.8331	Skaggs
TP-83	121	Subsurface	639023.8093	867919.0378	Skaggs
TP-84	122	Subsurface	638958.0463	868027.8000	Skaggs
TP-85	123	Subsurface	638979.5459	868158.0612	Skaggs
TP-86	124	Subsurface	638889.7538	868260.5000	Skaggs
TP-87	125	Subsurface	639027.6036	868324.9986	Skaggs
TP-88	126	Subsurface	639018.7506	868457.7893	Skaggs
TP-89	127	Subsurface	638902.4007	868555.1690	Skaggs
TP-90	128	Subsurface	638813.8734	867705.3079	Skaggs
TP-91	129	Subsurface	638696.2588	867701.5139	Skaggs
TP-92	130	Subsurface	638789.8445	867854.5395	Skaggs
TP-93	131	Subsurface	638678.5534	867921.5673	Skaggs

Table 3-1  
Sampling Locations  
Remedial Design  
Madison County Mines OU5  
Madison County, Missouri

Sample Location	Sample Number	Media	Northing	Easting	Comments
TP-94	132	Subsurface	638688.6705	868070.7990	Skaggs
TP-95	133	Subsurface	638783.5214	868146.6794	Skaggs
TP-96	134	Subsurface	638682.3474	868379.3794	Skaggs
TP-97	135	Subsurface	638770.8744	868279.4701	Skaggs
TP-98	136	Subsurface	638805.0207	868436.2897	Skaggs
TP-99	137	Subsurface	638724.0815	868556.4338	Skaggs
TP-100	138	Subsurface	638495.1755	867754.6303	Skaggs
TP-101	139	Subsurface	638590.0260	867697.7200	Skaggs
TP-102	140	Subsurface	638479.9994	867888.6858	Skaggs
TP-103	141	Subsurface	638588.7613	867872.2449	Skaggs
TP-104	142	Subsurface	638581.1734	868039.1819	Skaggs
TP-105	143	Subsurface	638461.0293	868131.5032	Skaggs
TP-106	144	Subsurface	638482.5289	868322.4691	Skaggs
TP-107	145	Subsurface	638611.5257	868287.0584	Skaggs
TP-108	146	Subsurface	638616.5844	868436.2897	Skaggs
TP-109	147	Subsurface	638586.2321	868555.1690	Skaggs
TP-110	148	Subsurface	638243.5054	867664.8385	Skaggs
TP-111	149	Subsurface	638353.5321	867747.0421	Skaggs
TP-112	150	Subsurface	638399.0602	867817.8641	Skaggs
TP-113	151	Subsurface	638414.2364	867927.8908	Skaggs
TP-114	152	Subsurface	638165.0955	867663.5737	Skaggs
TP-115	153	Subsurface	638041.1574	867611.7221	Skaggs
TP-116	154	Subsurface	637928.6012	867535.8417	Skaggs
TP-117	155	Subsurface	637840.0741	867457.4317	Skaggs
TP-118	156	Subsurface	637985.5118	867650.9271	Skaggs
TP-119	157	Subsurface	637872.9556	867591.4872	Skaggs
TP-120	158	Subsurface	637757.1606	867283.8258	Skaggs
TP-121	159	Subsurface	637773.0464	867379.0218	Skaggs
TP-122	160	Subsurface	637784.4286	867551.0175	Skaggs
<b>Sediment/Surface Water Samples</b>					
SED-1	52400	Sediment	641951.7247	866678.8336	Logtown Branch
SW-1	52400	Surface Water	641951.7247	866678.8336	Logtown Branch
SED-2	56400	Sediment	638520.0563	869373.1317	Eastern Tributary
SW-2	56400	Surface Water	638520.0563	869373.1317	Eastern Tributary
SED-3	57900	Sediment	638720.9231	866846.1393	Southern Tributary
SW-3	57900	Surface Water	638720.9231	866846.1393	Southern Tributary
SED-4	57901	Sediment	636397.1082	866298.3556	Southern Tributary
SW-4	57901	Surface Water	636397.1082	866298.3556	Southern Tributary
SED-5	57902	Sediment	633972.5596	866063.8072	Southern Tributary
SW-5	57902	Surface Water	633972.5596	866063.8072	Southern Tributary
SED-6	57903	Sediment	631932.4331	864870.8470	Southern Tributary
SW-6	57903	Surface Water	631932.4331	864870.8470	Southern Tributary
<b>Flood Plain Soils</b>					
FP-1	52301	Flood Plain Soils	641884.0952	866622.8583	North (Logtown Branch)
FP-2	52302	Flood Plain Soils	641873.7426	866640.1128	South (Logtown Branch)
FP-3	52303	Flood Plain Soils	642059.6224	866798.0984	North (Logtown Branch)
FP-4	52304	Flood Plain Soils	642043.0044	866809.4288	South (Logtown Branch)
FP-5	52305	Flood Plain Soils	642274.1929	866910.5290	North (Logtown Branch)
FP-6	52306	Flood Plain Soils	642263.3868	866927.5463	South (Logtown Branch)
FP-7	52307	Flood Plain Soils	642418.4413	867098.6939	North (Logtown Branch)

Table 3-1  
Sampling Locations  
Remedial Design  
Madison County Mines OU5  
Madison County, Missouri

Sample Location	Sample Number	Media	Northing	Easting	Comments
FP-8	52308	Flood Plain Soils	642401.1411	867108.7887	South (Logtown Branch)
FP-9	52309	Flood Plain Soils	642531.8994	867290.6952	North (Logtown Branch)
FP-10	52310	Flood Plain Soils	642516.4467	867303.3202	South (Logtown Branch)
FP-11	52311	Flood Plain Soils	642730.5535	867370.6140	West (Logtown Branch)
FP-12	52312	Flood Plain Soils	642736.5463	867389.7626	East (Logtown Branch)
FP-13	52313	Flood Plain Soils	642970.2548	867355.2777	West (Logtown Branch)
FP-14	52314	Flood Plain Soils	642964.1784	867374.3752	East (Logtown Branch)
FP-15	52315	Flood Plain Soils	643198.3636	867465.5905	West (Logtown Branch)
FP-16	52316	Flood Plain Soils	643184.6136	867479.9655	East (Logtown Branch)
FP-17	56301	Flood Plain Soils	638521.7870	869324.2577	North (Stream East of Skaggs)
FP-18	56302	Flood Plain Soils	638501.8038	869325.7581	South (Stream East of Skaggs)
FP-19	56303	Flood Plain Soils	638570.1508	869567.2622	North (Stream East of Skaggs)
FP-20	56304	Flood Plain Soils	638550.9451	869573.1050	South (Stream East of Skaggs)
FP-21	56305	Flood Plain Soils	638684.1577	869776.3149	North (Stream East of Skaggs)
FP-22	56306	Flood Plain Soils	638671.3303	869791.6662	South (Stream East of Skaggs)
FP-23	56307	Flood Plain Soils	638849.2063	869965.2148	North (Stream East of Skaggs)
FP-24	56308	Flood Plain Soils	638832.7336	869976.7230	South (Stream East of Skaggs)
FP-25	56309	Flood Plain Soils	638944.6575	870199.3810	North (Stream East of Skaggs)
FP-26	56310	Flood Plain Soils	638926.2508	870207.1621	South (Stream East of Skaggs)
FP-27	56311	Flood Plain Soils	639008.3267	870445.6262	North (Stream East of Skaggs)
FP-28	56312	Flood Plain Soils	638989.2641	870439.8972	South (Stream East of Skaggs)
FP-29	56313	Flood Plain Soils	639038.8040	870665.1402	North (Stream East of Skaggs)
FP-30	56314	Flood Plain Soils	639022.5540	870676.7025	South (Stream East of Skaggs)
FP-31	57701	Flood Plain Soils	638739.8423	866856.6209	West (Stream South of Catherine)
FP-32	57702	Flood Plain Soils	638735.2160	866876.3332	East (Stream South of Catherine)
FP-33	57703	Flood Plain Soils	638544.4759	866724.6383	West (Stream South of Catherine)
FP-34	57704	Flood Plain Soils	638527.7758	866735.6989	East (Stream South of Catherine)
FP-35	57705	Flood Plain Soils	638415.5959	866512.4458	West (Stream South of Catherine)
FP-36	57706	Flood Plain Soils	638397.7131	866521.6695	East (Stream South of Catherine)
FP-37	57707	Flood Plain Soils	638226.1639	866378.8808	West (Stream South of Catherine)
FP-38	57708	Flood Plain Soils	638220.3070	866398.0707	East (Stream South of Catherine)
FP-39	57709	Flood Plain Soils	638006.0761	866268.7284	West (Stream South of Catherine)
FP-40	57710	Flood Plain Soils	637999.6840	866287.8002	East (Stream South of Catherine)
FP-41	57711	Flood Plain Soils	637774.0500	866354.7610	West (Stream South of Catherine)
FP-42	57712	Flood Plain Soils	637778.6733	866374.2692	East (Stream South of Catherine)
FP-43	57713	Flood Plain Soils	637534.1499	866388.0379	West (Stream South of Catherine)
FP-44	57714	Flood Plain Soils	637532.2056	866408.0378	East (Stream South of Catherine)
FP-45	57715	Flood Plain Soils	637310.8682	866444.4761	West (Stream South of Catherine)
FP-46	57716	Flood Plain Soils	637320.6920	866461.8458	East (Stream South of Catherine)
FP-47	57717	Flood Plain Soils	637107.4427	866533.4188	West (Stream South of Catherine)
FP-48	57718	Flood Plain Soils	637097.5267	866551.4847	East (Stream South of Catherine)
FP-49	57719	Flood Plain Soils	636890.4090	866415.9568	West (Stream South of Catherine)
FP-50	57720	Flood Plain Soils	636890.5219	866435.8140	East (Stream South of Catherine)
FP-51	57721	Flood Plain Soils	636645.7832	866413.5434	West (Stream South of Catherine)
FP-52	57722	Flood Plain Soils	636641.2153	866433.1949	East (Stream South of Catherine)
FP-53	57723	Flood Plain Soils	636426.9388	866308.0449	West (Stream South of Catherine)
FP-54	57724	Flood Plain Soils	636416.1885	866325.0780	East (Stream South of Catherine)
FP-55	57725	Flood Plain Soils	636224.8753	866162.4543	West (Stream South of Catherine)
FP-56	57726	Flood Plain Soils	636218.1115	866181.2693	East (Stream South of Catherine)
FP-57	57727	Flood Plain Soils	636036.6548	866231.2488	West (Stream South of Catherine)

Table 3-1  
Sampling Locations  
Remedial Design  
Madison County Mines OU5  
Madison County, Missouri

Sample Location	Sample Number	Media	Northing	Easting	Comments
FP-58	57728	Flood Plain Soils	636037.4629	866251.3495	East (Stream South of Catherine)
FP-59	57729	Flood Plain Soils	635809.9834	866212.2046	West (Stream South of Catherine)
FP-60	57730	Flood Plain Soils	635805.5388	866232.3433	East (Stream South of Catherine)
FP-61	57731	Flood Plain Soils	635578.7332	866215.8155	West (Stream South of Catherine)
FP-62	57732	Flood Plain Soils	635578.1082	866235.9545	East (Stream South of Catherine)
FP-63	57733	Flood Plain Soils	635337.7957	866224.8781	West (Stream South of Catherine)
FP-64	57734	Flood Plain Soils	635331.8236	866244.0447	East (Stream South of Catherine)
FP-65	57735	Flood Plain Soils	635119.5389	866119.0335	West (Stream South of Catherine)
FP-66	57736	Flood Plain Soils	635120.2610	866139.2005	East (Stream South of Catherine)
FP-67	57737	Flood Plain Soils	634944.2971	865963.2697	West (Stream South of Catherine)
FP-68	57738	Flood Plain Soils	634929.0055	865976.1030	East (Stream South of Catherine)
FP-69	57739	Flood Plain Soils	634717.4117	865924.1901	West (Stream South of Catherine)
FP-70	57740	Flood Plain Soils	634719.7034	865944.1900	East (Stream South of Catherine)
FP-71	57741	Flood Plain Soils	634485.8978	866015.6515	West (Stream South of Catherine)
FP-72	57742	Flood Plain Soils	634491.1012	866035.0199	East (Stream South of Catherine)
FP-73	57743	Flood Plain Soils	634244.3923	866056.2863	West (Stream South of Catherine)
FP-74	57744	Flood Plain Soils	634245.0456	866076.2721	East (Stream South of Catherine)
FP-75	57745	Flood Plain Soils	633998.2983	866049.6134	West (Stream South of Catherine)
FP-76	57746	Flood Plain Soils	633999.8817	866069.5720	East (Stream South of Catherine)
FP-77	57747	Flood Plain Soils	633750.2844	866076.2524	West (Stream South of Catherine)
FP-78	57748	Flood Plain Soils	633751.4511	866096.3357	East (Stream South of Catherine)
FP-79	57749	Flood Plain Soils	633505.1396	866109.3012	West (Stream South of Catherine)
FP-80	57750	Flood Plain Soils	633511.1681	866128.4127	East (Stream South of Catherine)
FP-81	57751	Flood Plain Soils	633271.8369	866147.5754	West (Stream South of Catherine)
FP-82	57752	Flood Plain Soils	633268.3923	866167.4087	East (Stream South of Catherine)
FP-83	57753	Flood Plain Soils	633046.9597	866046.7699	West (Stream South of Catherine)
FP-84	57754	Flood Plain Soils	633044.8764	866066.8532	East (Stream South of Catherine)
FP-85	57755	Flood Plain Soils	632843.3528	865947.7402	West (Stream South of Catherine)
FP-86	57756	Flood Plain Soils	632833.4778	865965.1778	East (Stream South of Catherine)
FP-87	57757	Flood Plain Soils	632635.6239	865817.3930	West (Stream South of Catherine)
FP-88	57758	Flood Plain Soils	632625.4015	865835.2263	East (Stream South of Catherine)
FP-89	57759	Flood Plain Soils	632489.3158	865635.2494	North (Stream South of Catherine)
FP-90	57760	Flood Plain Soils	632469.7588	865639.9429	South (Stream South of Catherine)
FP-91	57761	Flood Plain Soils	632293.4225	865498.4140	North (Stream South of Catherine)
FP-92	57762	Flood Plain Soils	632277.0892	865509.9973	South (Stream South of Catherine)
FP-93	57763	Flood Plain Soils	632189.5073	865287.6388	North (Stream South of Catherine)
FP-94	57764	Flood Plain Soils	632170.7000	865294.3865	South (Stream South of Catherine)
FP-95	57765	Flood Plain Soils	632038.5529	865096.5851	North (Stream South of Catherine)
FP-96	57766	Flood Plain Soils	632021.3446	865106.7934	South (Stream South of Catherine)
FP-97	57767	Flood Plain Soils	631937.4127	864871.6338	North (Stream South of Catherine)
FP-98	57768	Flood Plain Soils	631918.5584	864878.5088	South (Stream South of Catherine)



Table 3-2  
Primary Sample Requirements  
Remedial Design  
Madison County Mines OU5  
Madison County, Missouri

Sample Location	Sample Number	Media	Number of Samples	Sample Type	Sample Depth	Analytical Parameters			
						Lead	RCRA Metals (excluding Hg), plus Co, Mn, Ni	Dissolved Metals	Other Water Parameters
Surface Samples									
SS-1 to SS-48	28200 to 28247	Surface soil	1	Discrete	Surface	All samples (XRF); 10% of sample locations to EPA lab	Yes	No	No
SS-49 to SS-105	30200 to 30256	Surface soil	1		Discrete	Surface	All samples (XRF); 10% of sample locations to EPA lab	Yes	No
Subsurface Test Pit Samples									
TP-1 to TP-61	1 to 61	Surface/Subsurface	Depends on Thickness	Discrete Interval	Consecutive 1 ft intervals	All samples (XRF); 10% of sample locations to EPA lab	Yes	No	No
TP-100 to TP-160	100 to 160	Surface/Subsurface	Depends on Thickness	Discrete Interval	Consecutive 1 ft intervals	All samples (XRF); 10% of sample locations to EPA lab	Yes	No	No
FloodPlain Soils									
FP-1 to FP-16	52301 to 52316	Floodplain Soil	Depends on Thickness	Discrete Interval	surface sample; 1 ft. intervals at one location on each side of stream	All samples (XRF)	No	No	No
FP-17 to FP-30	56301 to 56314	Floodplain Soil	Depends on Thickness	Discrete Interval	surface sample; 1 ft. intervals at one location on each side of stream	All samples (XRF)	No	No	No
FP-31 to FP-98	57701 to 57768	Floodplain Soil	Depends on Thickness	Discrete Interval	surface sample; 1 ft. intervals at one location on each side of stream	All samples (XRF)	No	No	No

Table 3-2  
Primary Sample Requirements  
Remedial Design  
Madison County Mines OU5  
Madison County, Missouri

Sample Location	Sample Number	Media	Number of Samples	Sample Type	Sample Depth	Analytical Parameters			
						Lead	RCRA Metals (excluding Hg), plus Co, Mn, Ni	Dissolved Metals	Other Water Parameters
Surface Water Samples									
SW-1	52400	Surface Water	1	Discrete	n/a	All samples (Lab)	Yes, Excluding Co and Mn	Yes	Yes
SW-2	56400	Surface Water	1	Discrete	n/a	All samples (Lab)	Yes, Excluding Co and Mn	Yes	Yes
SW-3 to SW-6	57900 to 57904	Surface Water	1	Discrete	n/a	All samples (Lab)	Yes, Excluding Co and Mn	Yes	Yes
Sediment Samples									
SED-1	52400	Sediment	1	Discrete	n/a	All samples (Lab)	Yes, Excluding Co and Mn	No	No
SED-2	56400	Sediment	1	Discrete	n/a	All samples (Lab)	Yes, Excluding Co and Mn	No	No
SED-3 to SED-6	57900 to 57904	Sediment	1	Discrete	n/a	All samples (Lab)	Yes, Excluding Co and Mn	No	No
Groundwater									
To Be Determined	28011 to 28014	Groundwater	1.0	Discrete	n/a	All samples (Lab)	Yes, Excluding Co and Ni	Yes	Yes
To Be Determined	30011 to 30014	Groundwater	1.0	Discrete	n/a	All samples (Lab)	Yes, Excluding Co and Ni	Yes	Yes



Table 3-3  
QA/QC Sample Requirements  
Madison County Mines OU5 Remedial Design  
Madison County, Missouri

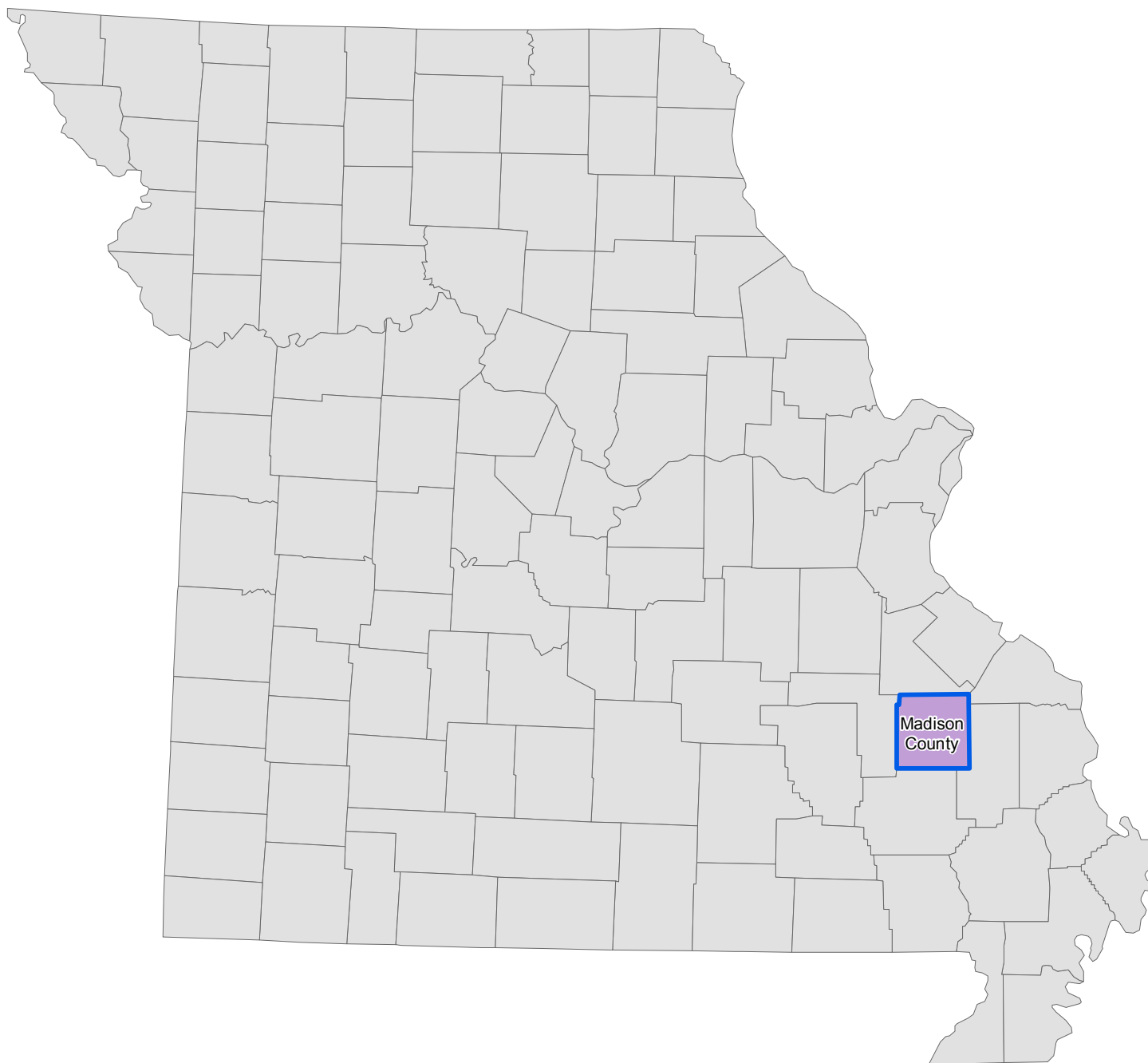
Environmental Medium	No. of Primary Analytical Samples	No. Confirmation Samples	No. of Field Duplicates	Matrix Spike/ Matrix Spike Duplicate	No. of Equipment Rinsates
Surface	105	11	0	1	1
Surface/Subsurface	122	13	0	1	1
Flood Plain Soil	98	0	0	0	0
Surface Water*	6	0	1	1	0
Sediment*	6	0	1	1	1
Groundwater**	8	0	1	1	1

\*Sediment and Surface Water Samples will be taken at the same locations. \*\*Groundwater locations will be determined after the repository has been completed.



## FIGURES





0 12.5 25 50

Miles

1" = 47 miles

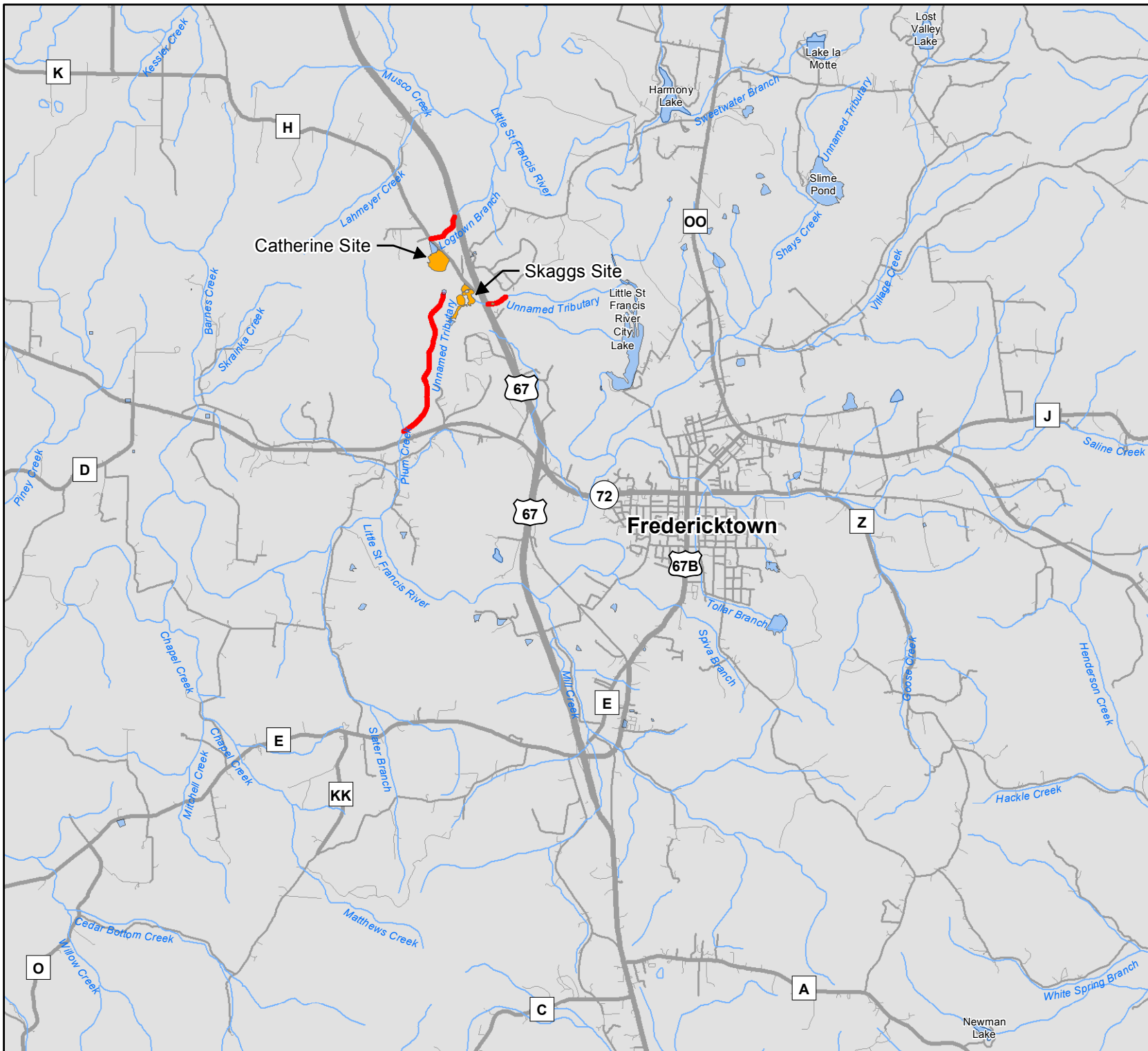
## Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

**Figure 1-1**  
Location Map







### Legend

- Mine Waste OU5 (Catherine & Skaggs Sites)
- Lakes
- Streams to be Sampled
- Streams
- Local Road
- County Road
- State Highway
- US Highway



0 1,750 3,500 7,000  
Feet  
1" = 7,000'

### Field Sampling Plan

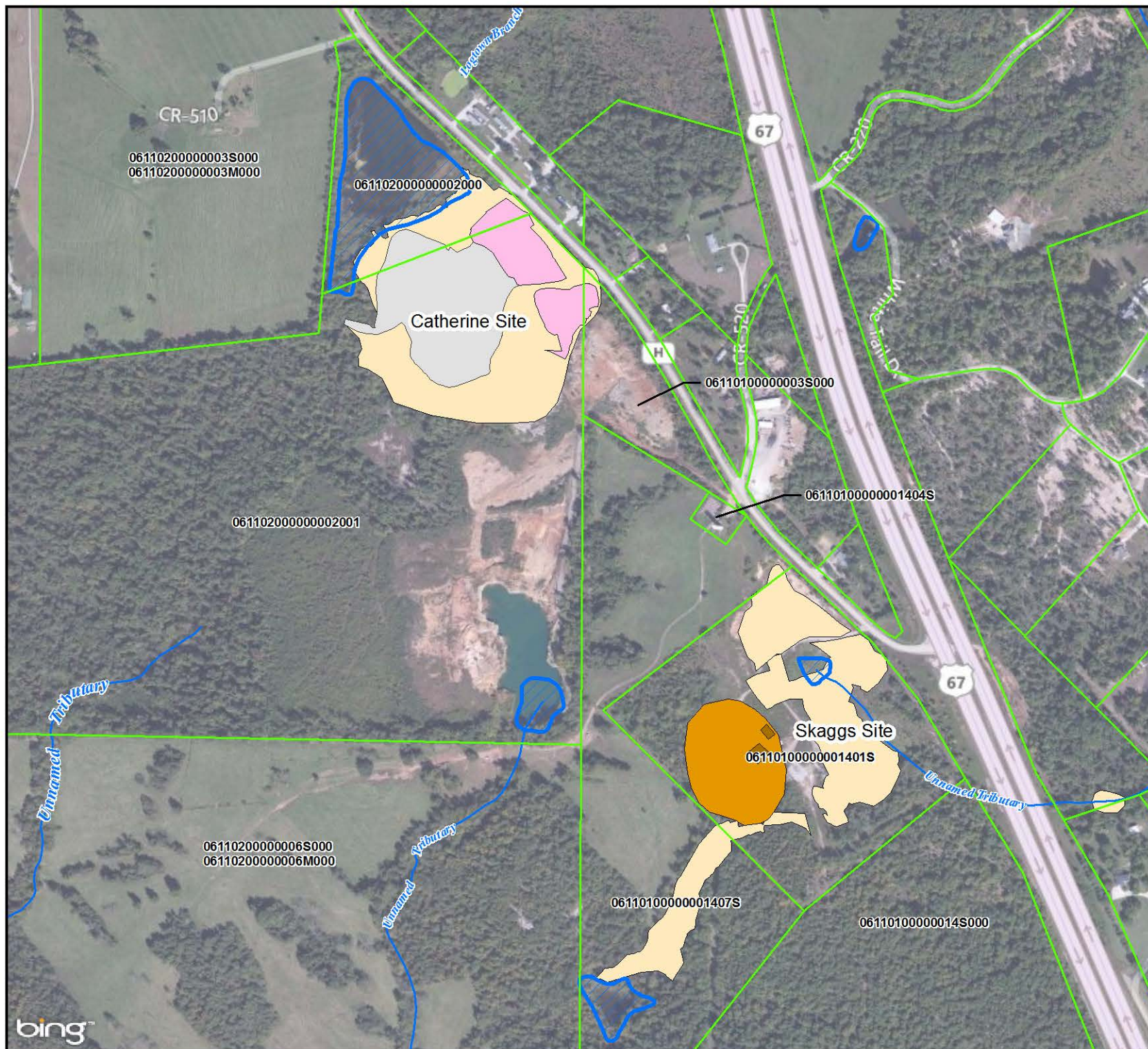
Madison County Mines  
Fredericktown, Missouri

**Figure 1-2**  
Site Map









### Legend

- Chat
- Clean Soil Over Chat
- Excavated Yard Soil Over Chat
- Sawdust pile
- Shop Building
- Lakes
- Parcels



0 150 300 600  
Feet

1" = 600'

### Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

### Figure 1-3

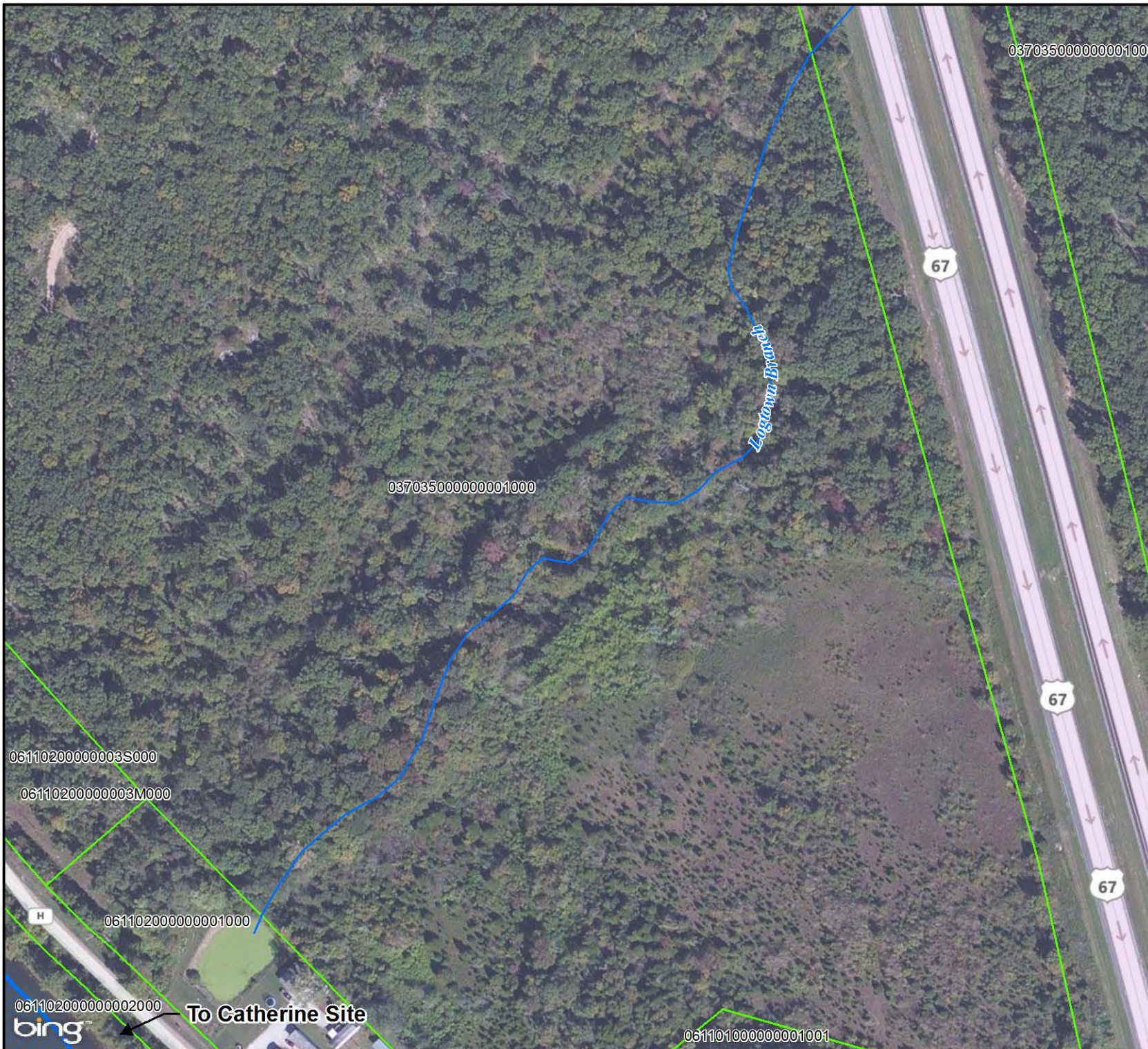
Features of the Catherine  
Mine and Skaggs Chat Area



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### Legend

- Streams
- Lakes
- Parcels



0 50 100 200  
Feet

1" = 200'

### Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

### Figure 1-4

Features of Logtown Branch

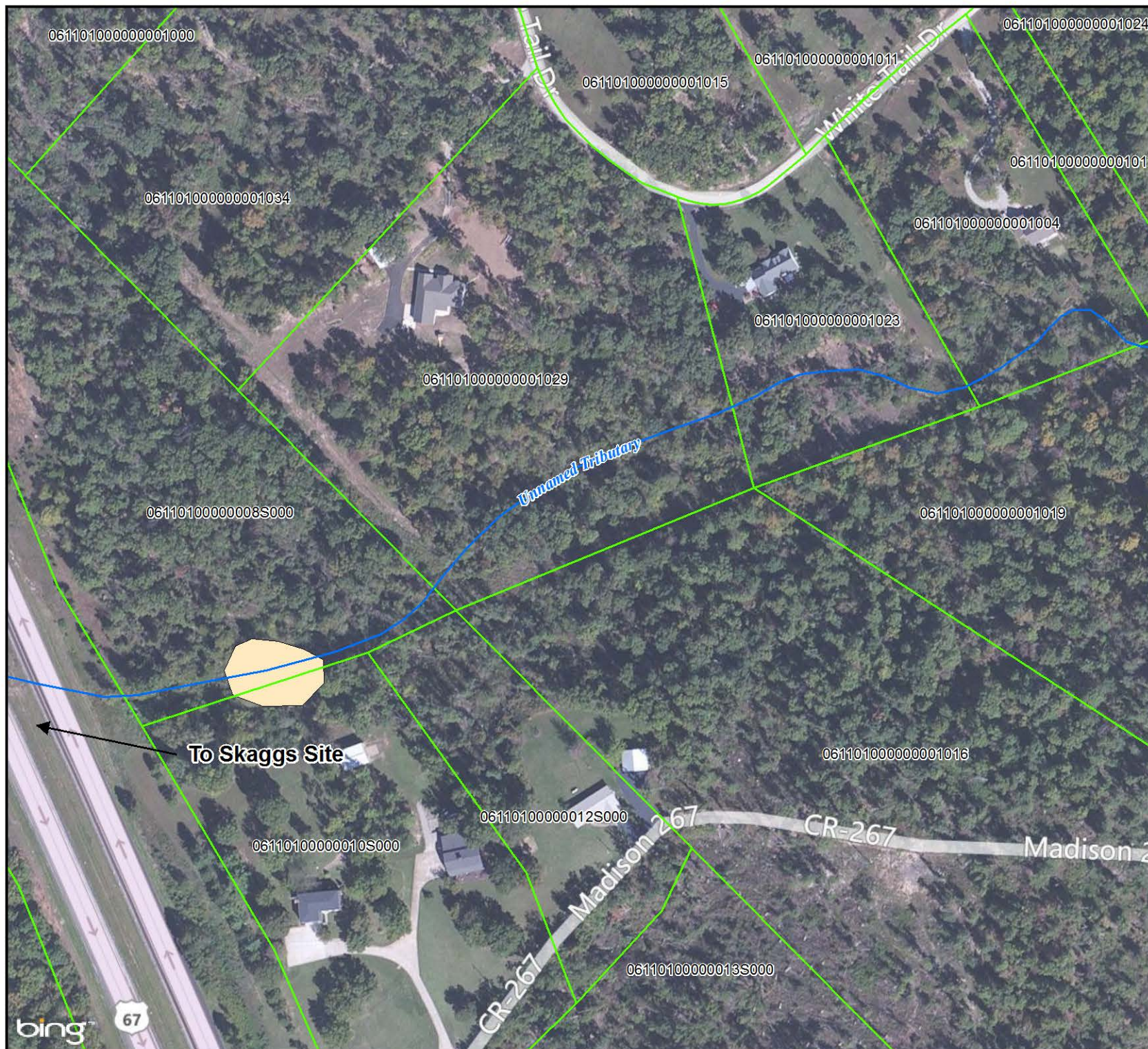


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### Legend

- Streams
- Chat
- Parcels



0 50 100 200  
Feet

1" = 200'

### Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

### Figure 1-5

Features of Intermittent Stream  
East of Skaggs Site

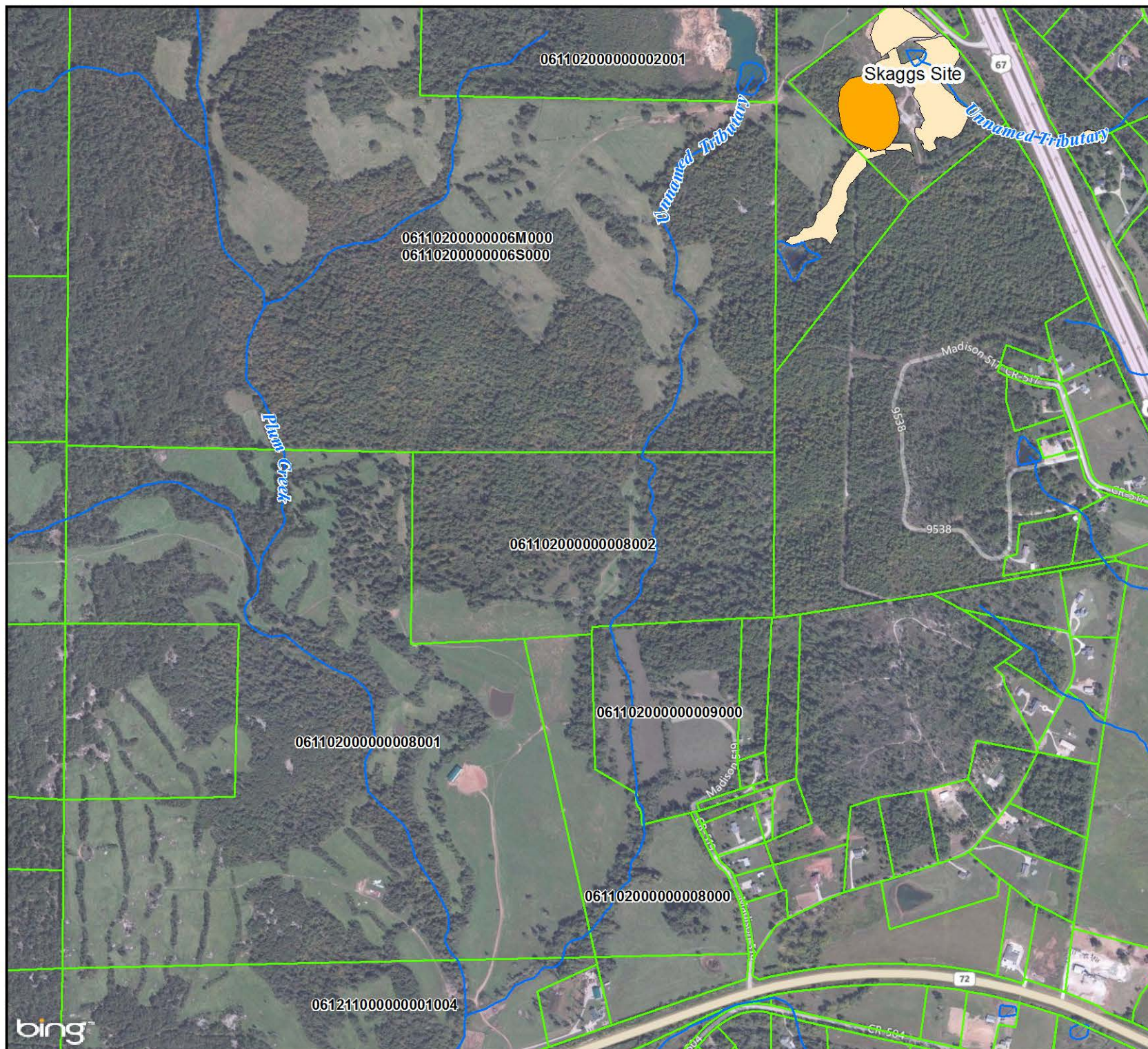


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### Legend

- Streams
- Chat
- Sawdust pile
- Lakes
- Parcels



0 250 500 1,000  
Feet

1" = 1,000'

### Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

### Figure 1-6

Features of Intermittent Stream  
South of the Catherine Sub Site

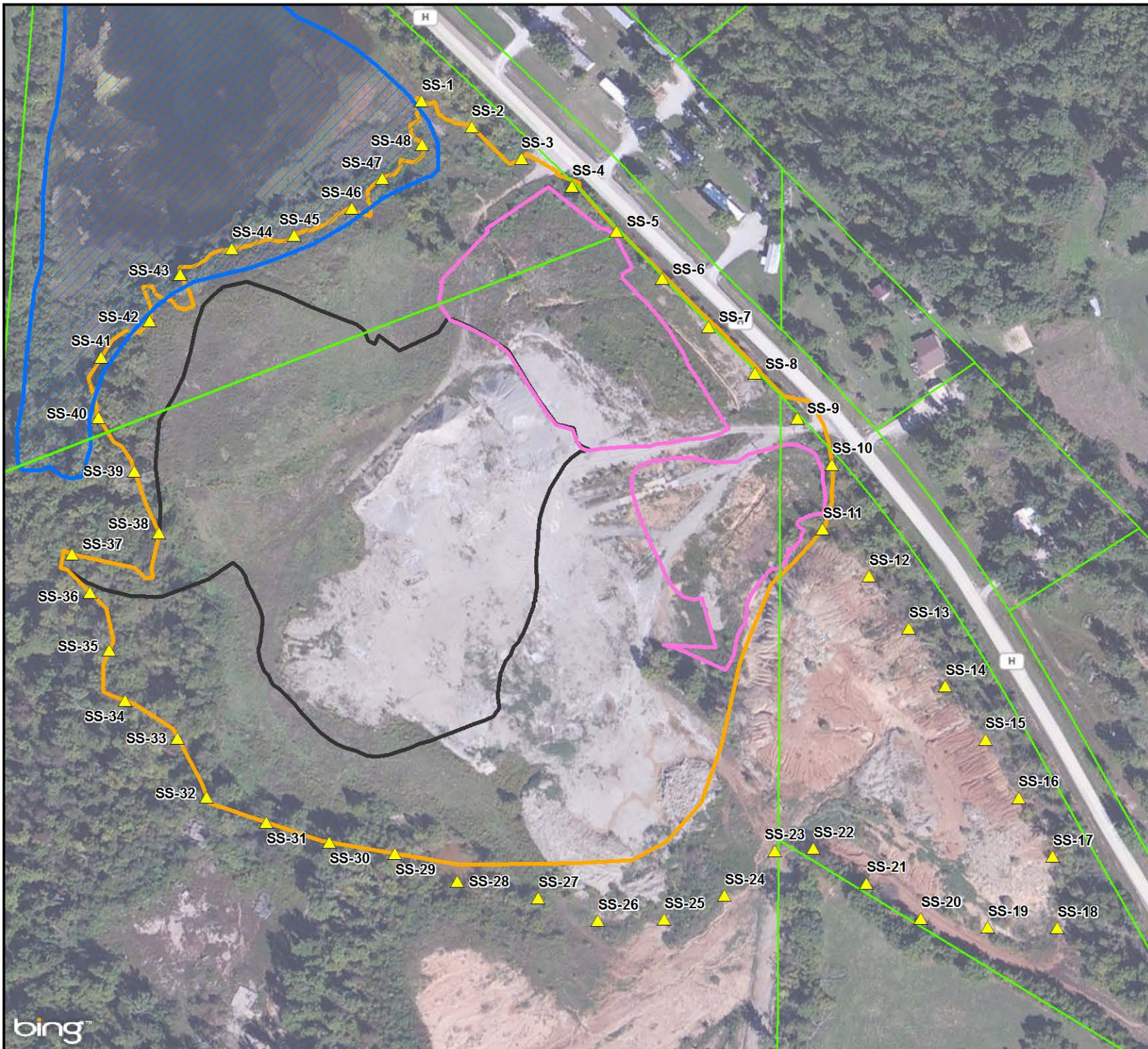


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### Legend

- ▲ Surface Soil Sample
- Chat
- Clean Soil Over Chat
- Excavated Yard Soil Over Chat
- Lakes
- Parcels



0 50 100 200  
Feet

1" = 200'

### Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

### Figure 3-1

Catherine Sub Site  
Surface Soil Samples









### Legend

- Surface Soil Sample
- Streams
- Chat
- Sawdust pile
- Lakes
- Parcels



0 75 150 300  
Feet

1" = 300'

### Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

### Figure 3-2

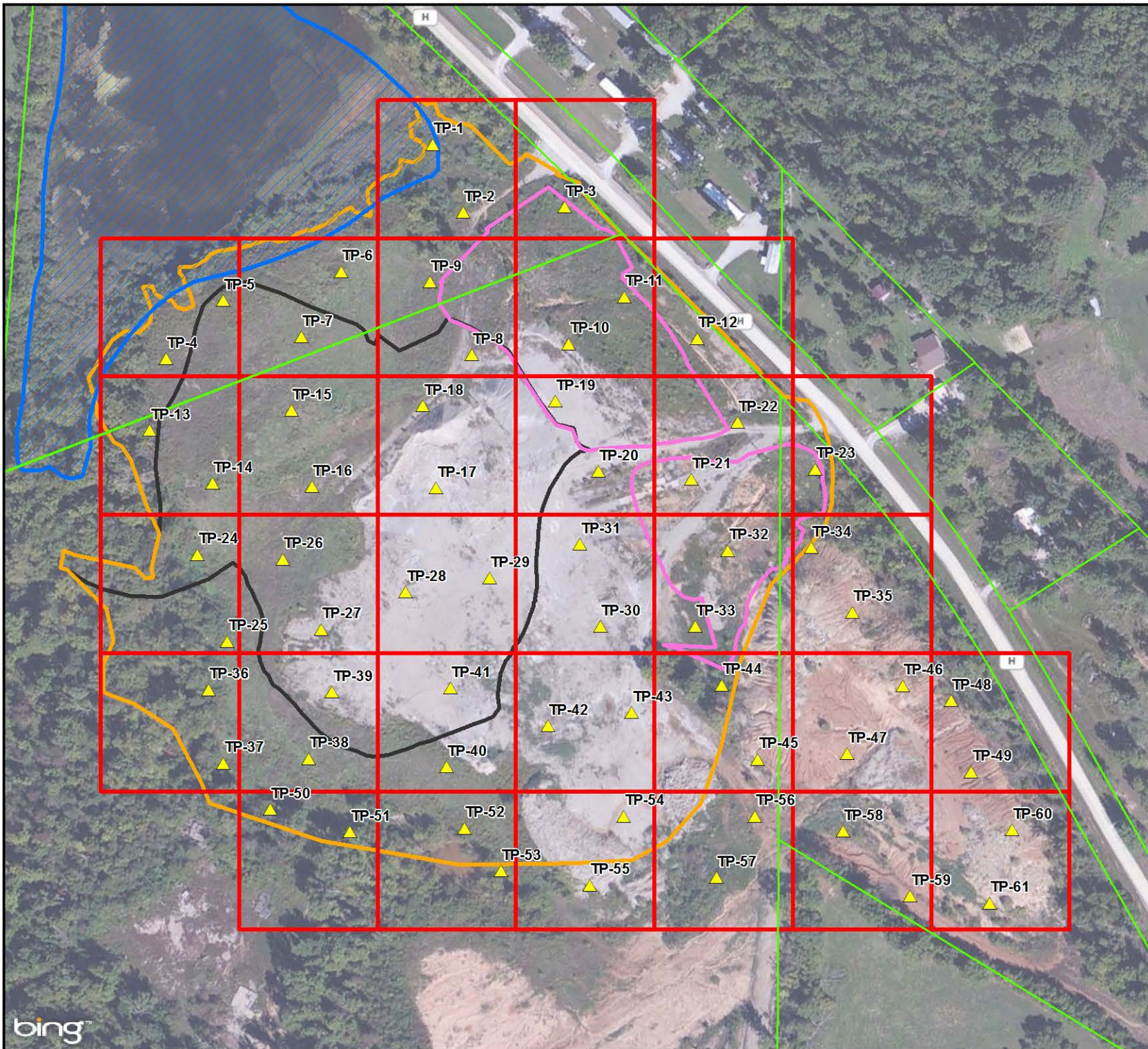
Skaggs Sub Site  
Surface Soil Samples



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### Legend

- Surface/Subsurface Soil Sample
- Catherine Site Sample Grid (1 acre cells)
- Chat
- Clean Soil Over Chat
- Excavated Yard Soil Over Chat
- Lakes
- Parcels



0 50 100 200  
Feet

1" = 200'

### Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

### Figure 3-3

Catherine Sub Site

Surface-Subsurface Soil Samples

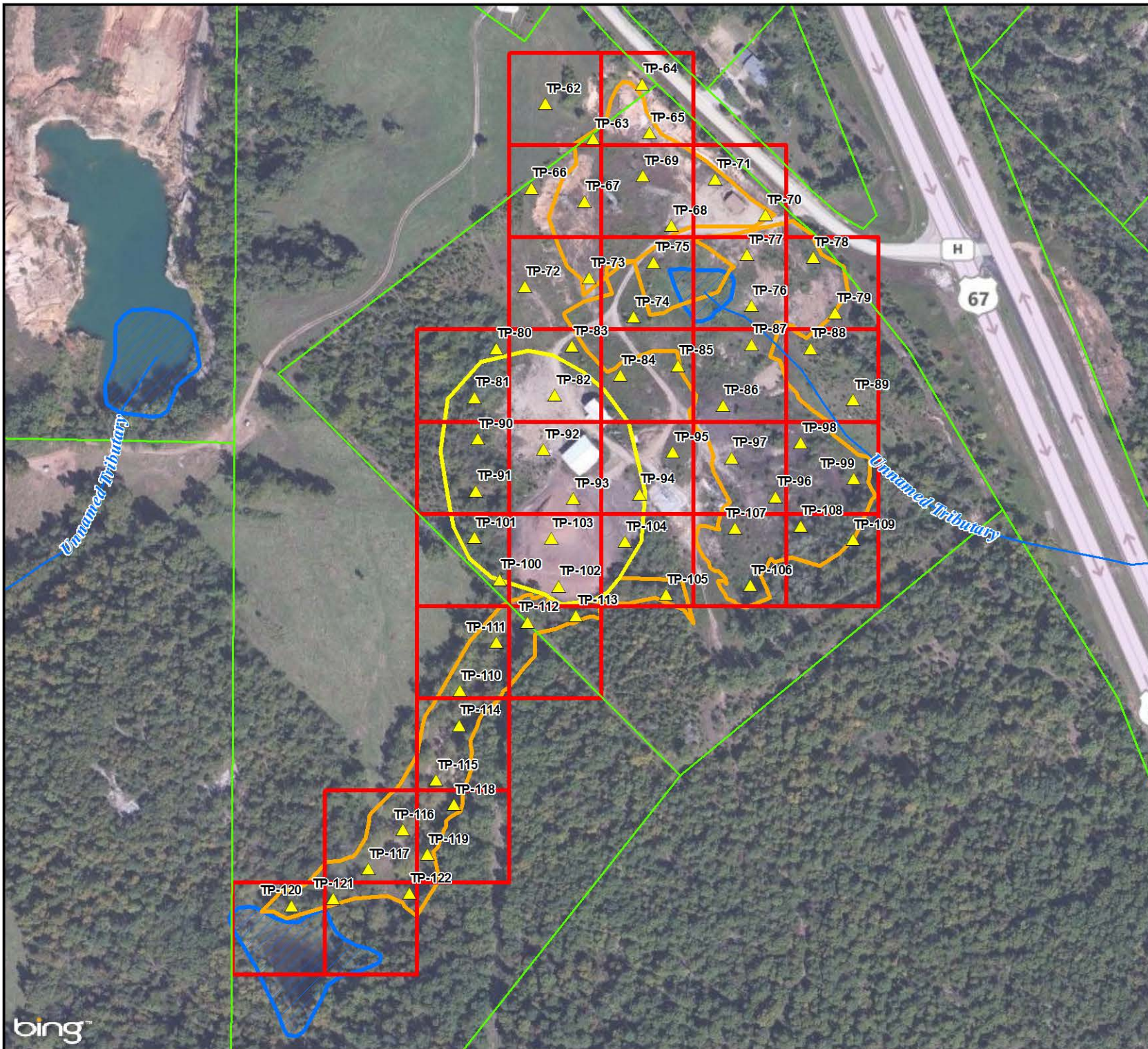


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### Legend

- Surface/Subsurface Soil Sample
- Streams
- Skaggs Site Sample Grid (1 acre cells)
- Chat
- Sawdust pile
- Lakes
- Parcels



0 75 150 300  
Feet

1" = 300'

### Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

### Figure 3-4

Skaggs Sub Site

Surface-Subsurface Soil Samples

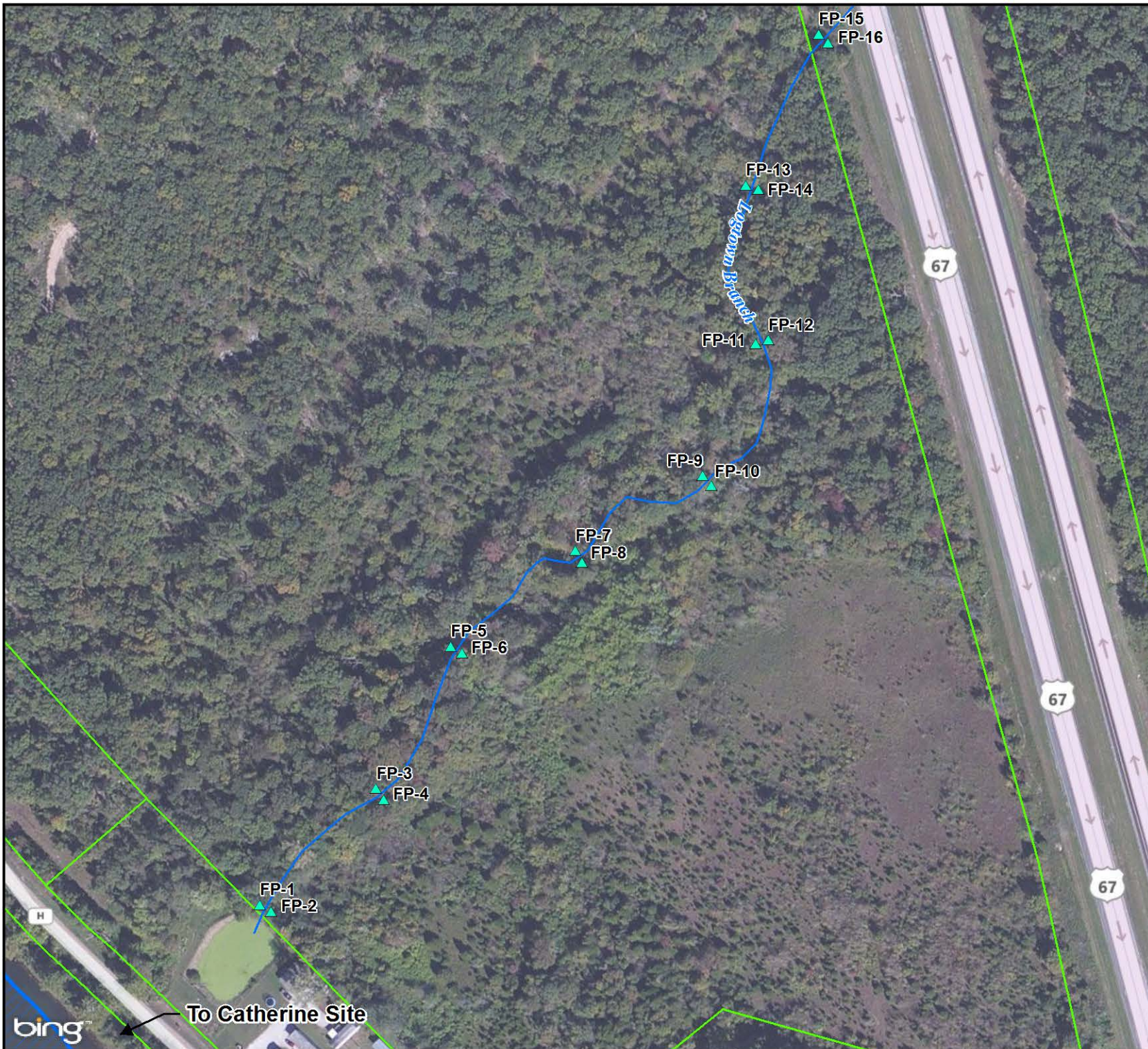


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### Legend

-  Floodplain Soil Sample
-  Streams
-  Lakes
-  Parcels



0 50 100 200  
Feet

1" = 200'

### Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

### Figure 3-5

Logtown Branch  
Flood Plain Soil Samples

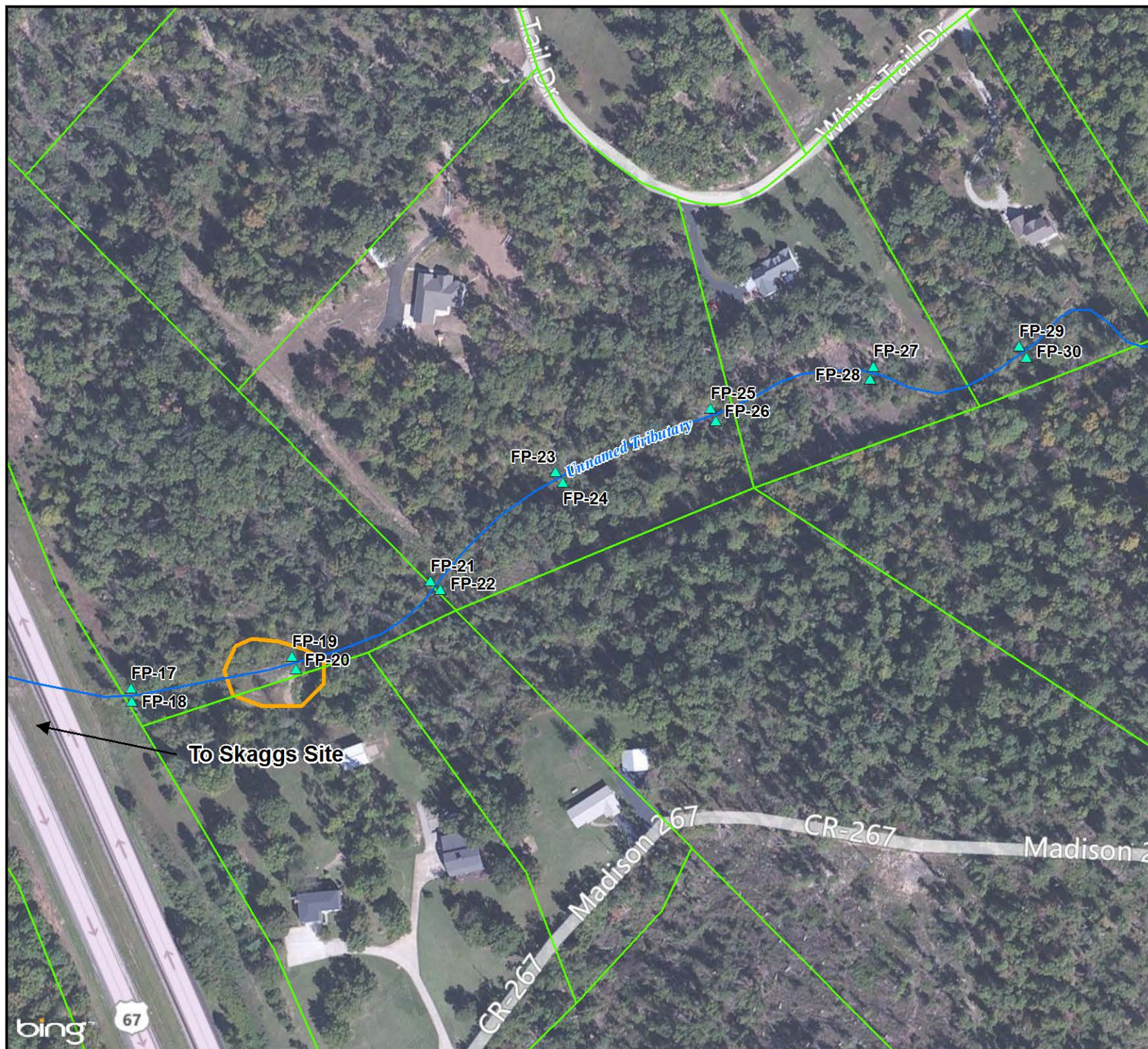


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### Legend

- Floodplain Soil Sample
- Streams
- Chat
- Parcels



0 50 100 200  
Feet

1" = 200'

### Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

### Figure 3-6

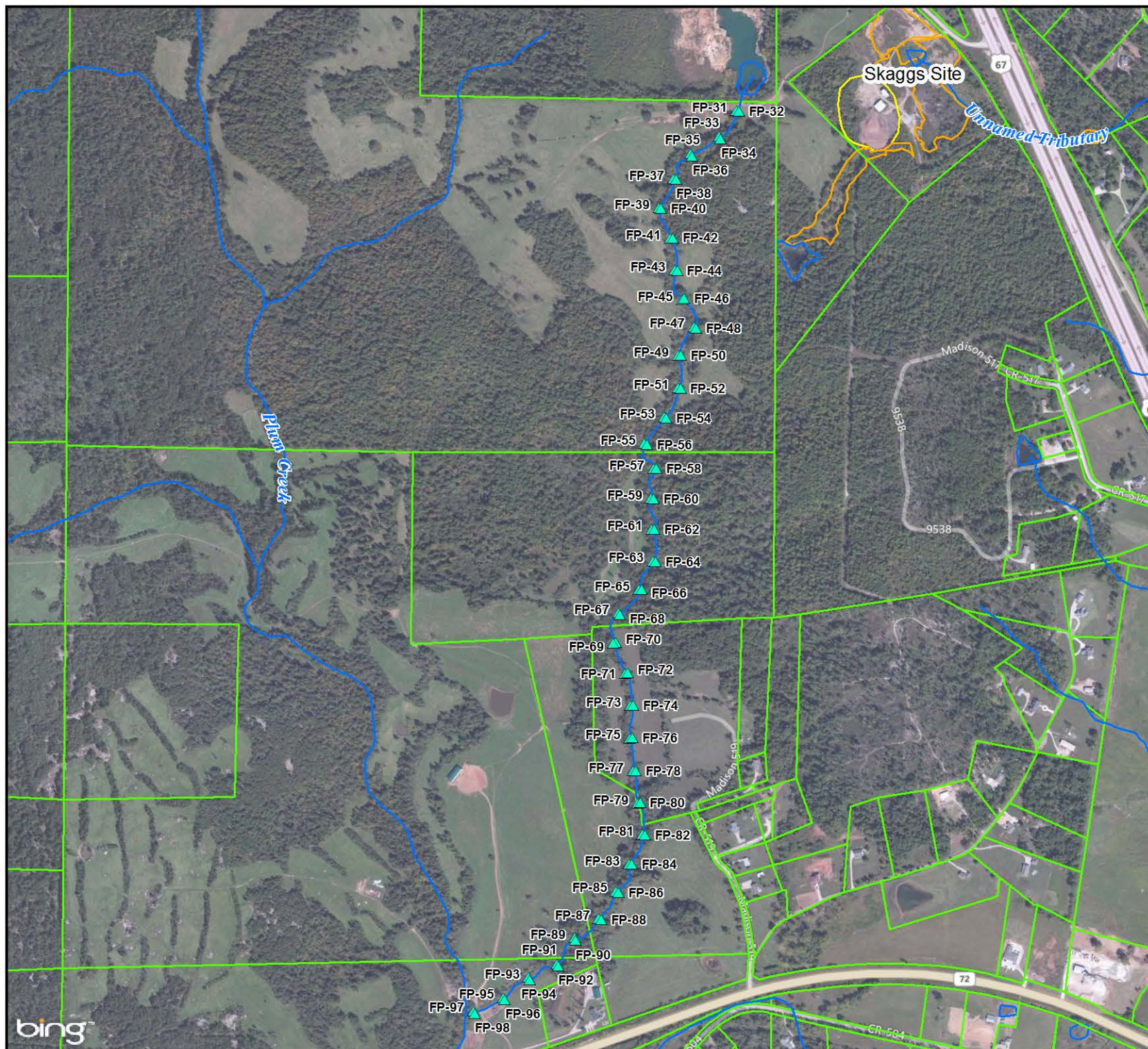
Intermittent Stream East  
of Skaggs Sub Site  
Flood Plain Soil Samples











### Legend

- Floodplain Soil Sample
- Streams
- Chat
- Sawdust pile
- Lakes
- Parcels



0 250 500 1,000  
Feet

1" = 1,000'

### Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

### Figure 3-7

Intermittent Stream South of  
the Catherine Sub Site  
Flood Plain Soil Samples

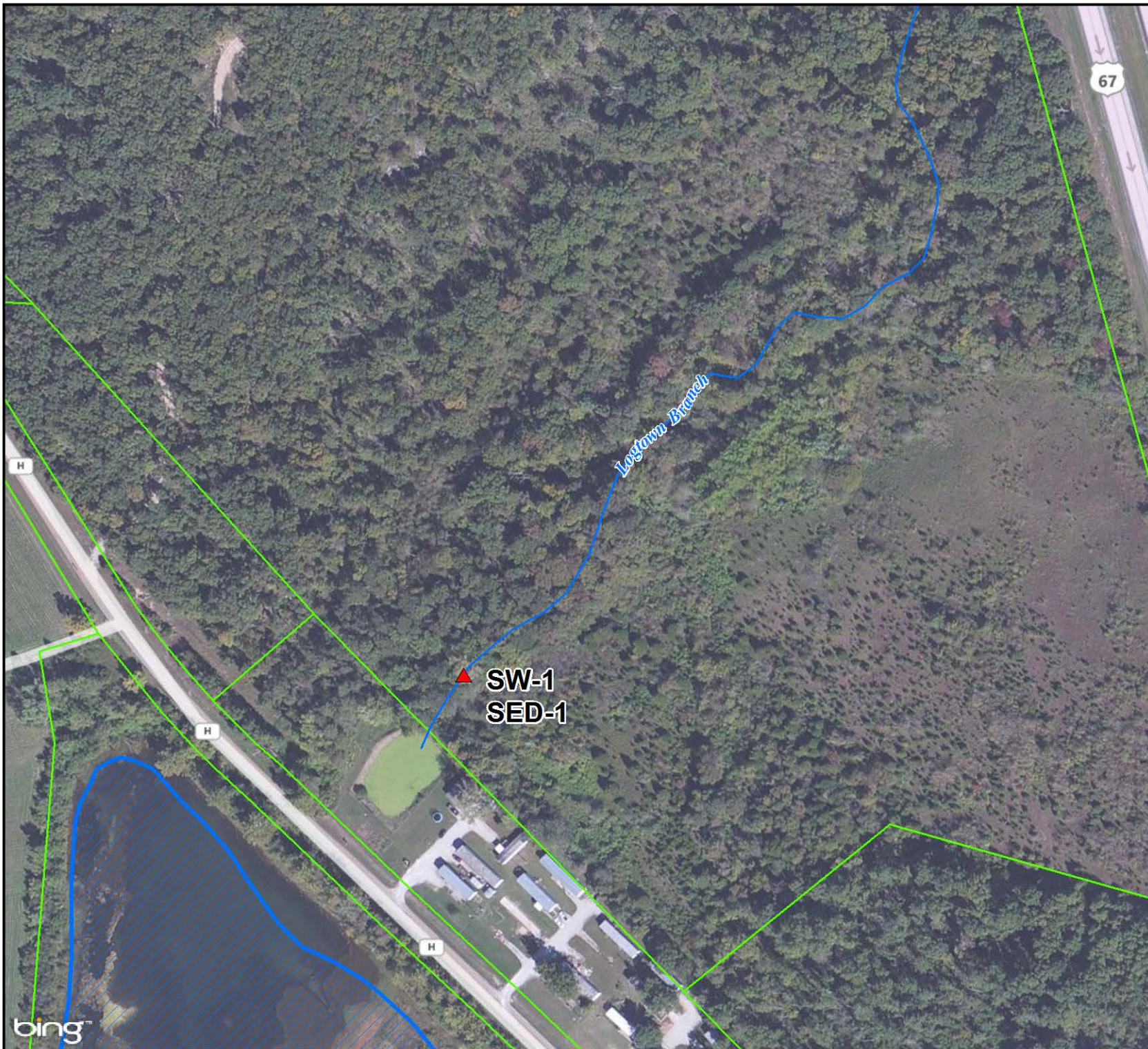


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### Legend

-  Sediment/Surface Water Sample
-  Streams
-  Lakes
-  Parcels



0 50 100 200  
Feet

1" = 200'

### Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

### Figure 3-8

Logtown Branch

Sediment and Surface Water Samples



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### Legend

-  Sediment/Surface Water Sample
-  Streams
-  Chat
-  Parcels



0 25 50 100  
Feet

1" = 100'

### Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

### Figure 3-9

Intermittent Stream East of  
the Skaggs Sub Site

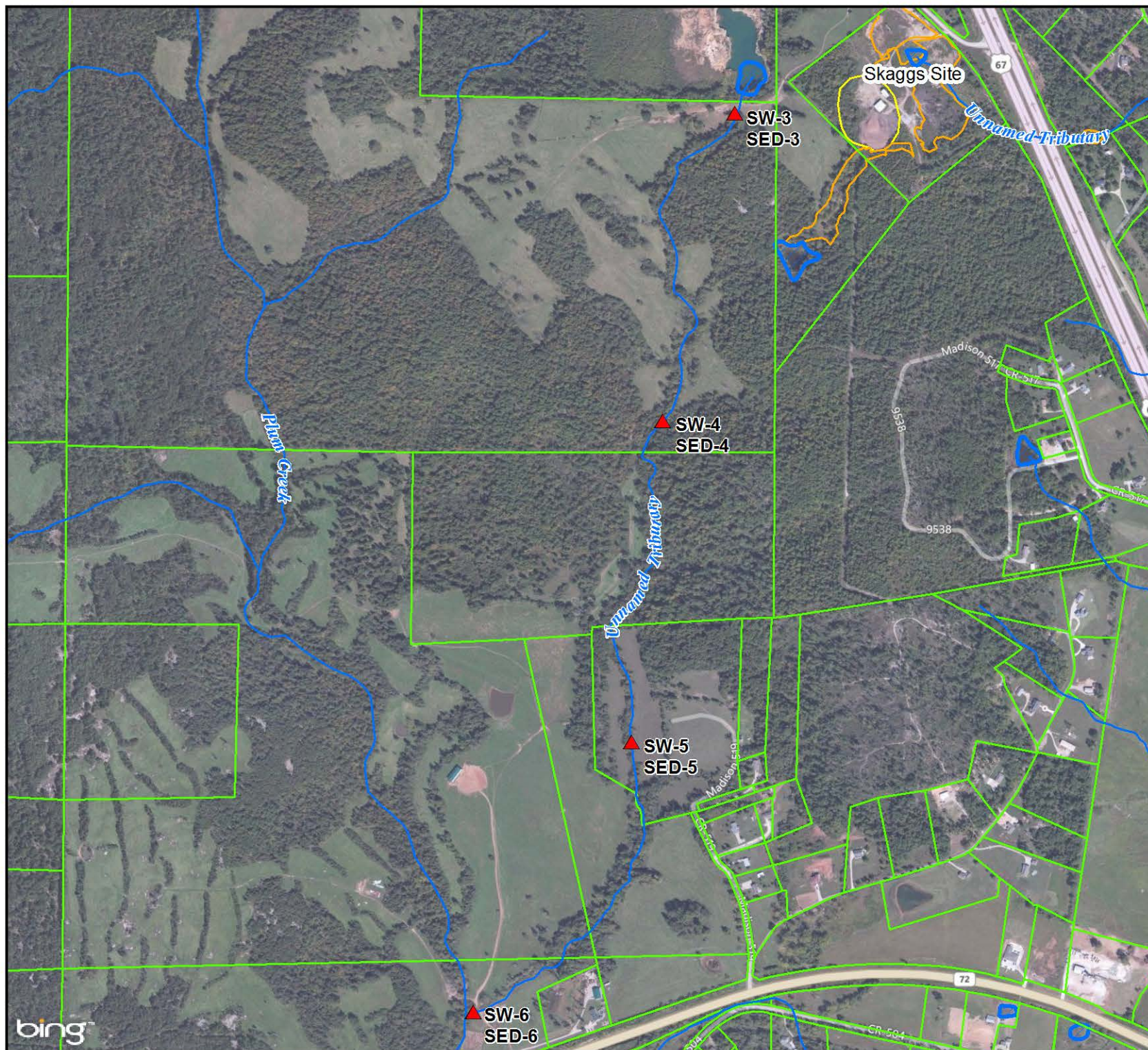
Sediment and Surface Water Samples



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### Legend

- ▲ Sediment/Surface Water Sample
- ~ Streams
- Chat
- Sawdust pile
- Lakes
- Parcels



0 250 500 1,000  
Feet

1" = 1,000'

### Field Sampling Plan

Madison County Mines  
Fredericktown, Missouri

### Figure 3-10

Intermittent Stream South of  
the Catherine Sub Site

Sediment and Surface Water Samples



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## Appendix A



EPA Region 7  
Standard Operating Procedure  
No. 2012  
Soil Sampling  
February 18, 2000







# U. S. EPA ENVIRONMENTAL RESPONSE TEAM

## STANDARD OPERATING PROCEDURES

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### SOIL SAMPLING

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3.0	SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE
4.0	POTENTIAL PROBLEMS
5.0	EQUIPMENT
6.0	REAGENTS
7.0	PROCEDURES
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7.2	Sample Collection
7.2.1	Surface Soil Samples
7.2.2	Sampling at Depth with Augers and Thin Wall Tube Samplers
7.2.3	Sampling at Depth with a Trier
7.2.4	Sampling at Depth with a Split Spoon (Barrel) Sampler
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8.0	CALCULATIONS
9.0	QUALITY ASSURANCE/QUALITY CONTROL
10.0	DATA VALIDATION
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## STANDARD OPERATING PROCEDURES

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### SOIL SAMPLING

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#### 1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to describe the procedures for the collection of representative soil samples. Sampling depths are assumed to be those that can be reached without the use of a drill rig, direct-push, or other mechanized equipment (except for a back-hoe). Analysis of soil samples may determine whether concentrations of specific pollutants exceed established action levels, or if the concentrations of pollutants present a risk to public health, welfare, or the environment.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the actual procedures used should be documented and described in an appropriate site report.

Mention of trade names or commercial products does not constitute U.S. Environmental Protection Agency (EPA) endorsement or recommendation for use.

#### 2.0 METHOD SUMMARY

Soil samples may be collected using a variety of methods and equipment depending on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type. Near-surface soils may be easily sampled using a spade, trowel, and scoop. Sampling at greater depths may be performed using a hand auger, continuous flight auger, a trier, a split-spoon, or, if required, a backhoe.

#### 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Chemical preservation of solids is not generally recommended. Samples should, however, be cooled and protected from sunlight to minimize any potential reaction. The amount of sample to be collected and proper sample container type are discussed in ERT/REAC SOP #2003 Rev. 0.0 08/11/94, *Sample Storage, Preservation and Handling*.

#### 4.0 INTERFERENCES AND POTENTIAL PROBLEMS

There are two primary potential problems associated with soil sampling - cross contamination of samples and improper sample collection. Cross contamination problems can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection can involve using contaminated equipment, disturbance of the matrix resulting in compaction of the sample, or inadequate homogenization of the samples where required, resulting in variable, non-representative results.

#### 5.0 EQUIPMENT



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### SOIL SAMPLING

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Soil sampling equipment includes the following:

- Maps/plot plan
- Safety equipment, as specified in the site-specific Health and Safety Plan
- Survey equipment or global positioning system (GPS) to locate sampling points
- Tape measure
- Survey stakes or flags
- Camera and film
- Stainless steel, plastic, or other appropriate homogenization bucket, bowl or pan
- Appropriate size sample containers
- Ziplock plastic bags
- Logbook
- Labels
- Chain of Custody records and custody seals
- Field data sheets and sample labels
- Cooler(s)
- Ice
- Vermiculite
- Decontamination supplies/equipment
- Canvas or plastic sheet
- Spade or shovel
- Spatula
- Scoop
- Plastic or stainless steel spoons
- Trowel(s)
- Continuous flight (screw) auger
- Bucket auger
- Post hole auger
- Extension rods
- T-handle
- Sampling trier
- Thin wall tube sampler
- Split spoons
- Vehimeyer soil sampler outfit
  - Tubes
  - Points
  - Drive head
  - Drop hammer
  - Puller jack and grip
- Backhoe



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### SOIL SAMPLING

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Reagents are not used for the preservation of soil samples. Decontamination solutions are specified in ERT/REAC SOP #2006 Rev. 0.0 08/11/94, *Sampling Equipment Decontamination*, and the site specific work plan.

#### 7.0 PROCEDURES

##### 7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required.
2. Obtain necessary sampling and monitoring equipment.
3. Decontaminate or pre-clean equipment, and ensure that it is in working order.
4. Prepare schedules and coordinate with staff, client, and regulatory agencies, if appropriate.
5. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety Plan.
6. Use stakes, flagging, or buoys to identify and mark all sampling locations. Specific site factors, including extent and nature of contaminant, should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All staked locations should be utility-cleared by the property owner or the On-Scene-Coordinator (OSC) prior to soil sampling; and utility clearance should always be confirmed before beginning work.

##### 7.2 Sample Collection

###### 7.2.1 Surface Soil Samples

Collection of samples from near-surface soil can be accomplished with tools such as spades, shovels, trowels, and scoops. Surface material is removed to the required depth and a stainless steel or plastic scoop is then used to collect the sample.

This method can be used in most soil types but is limited to sampling at or near the ground surface. Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sample team member. A flat, pointed mason trowel to cut a block of the desired soil is helpful when undisturbed profiles are required. Tools plated with chrome or other materials should not be used. Plating is particularly common with garden implements such as potting trowels.

The following procedure is used to collect surface soil samples:



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### SOIL SAMPLING

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1. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.
2. Using a pre-cleaned, stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade.
3. If volatile organic analysis is to be performed, transfer the sample directly into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval or location into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

#### 7.2.2 Sampling at Depth with Augers and Thin Wall Tube Samplers

This system consists of an auger, or a thin-wall tube sampler, a series of extensions, and a "T" handle (Figure 1, Appendix A). The auger is used to bore a hole to a desired sampling depth, and is then withdrawn. The sample may be collected directly from the auger. If a core sample is to be collected, the auger tip is then replaced with a thin wall tube sampler. The system is then lowered down the borehole, and driven into the soil to the completion depth. The system is withdrawn and the core is collected from the thin wall tube sampler.

Several types of augers are available; these include: bucket type, continuous flight (screw), and post-hole augers. Bucket type augers are better for direct sample recovery because they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the flights. The continuous flight augers are satisfactory when a composite of the complete soil column is desired. Post-hole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy soil and cannot be used below a depth of approximately three feet.

The following procedure is used for collecting soil samples with the auger:

1. Attach the auger bit to a drill rod extension, and attach the "T" handle to the drill rod.



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### SOIL SAMPLING

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2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first three to six inches of surface soil for an area approximately six inches in radius around the drilling location.
3. Begin augering, periodically removing and depositing accumulated soils onto a plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole, and avoids possible contamination of the surrounding area.
4. After reaching the desired depth, slowly and carefully remove the auger from the hole. When sampling directly from the auger, collect the sample after the auger is removed from the hole and proceed to Step 10.
5. Remove auger tip from the extension rods and replace with a pre-cleaned thin wall tube sampler. Install the proper cutting tip.
6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into the soil. Do not scrape the borehole sides. Avoid hammering the rods as the vibrations may cause the boring walls to collapse.
7. Remove the tube sampler, and unscrew the drill rods.
8. Remove the cutting tip and the core from the device.
9. Discard the top of the core (approximately 1 inch), as this possibly represents material collected before penetration of the layer of concern. Place the remaining core into the appropriate labeled sample container. Sample homogenization is not required.
10. If volatile organic analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly.

When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.



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11. If another sample is to be collected in the same hole, but at a greater depth, reattach the auger bit to the drill and assembly, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.
12. Abandon the hole according to applicable state regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

#### 7.2.3 Sampling with a Trier

The system consists of a trier, and a "T" handle. The auger is driven into the soil to be sampled and used to extract a core sample from the appropriate depth.

The following procedure is used to collect soil samples with a sampling trier:

1. Insert the trier (Figure 2, Appendix A) into the material to be sampled at a 0° to 45° angle from horizontal. This orientation minimizes the spillage of sample.
2. Rotate the trier once or twice to cut a core of material.
3. Slowly withdraw the trier, making sure that the slot is facing upward.
4. If volatile organic analyses are required, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

#### 7.2.4 Sampling at Depth with a Split Spoon (Barrel) Sampler

Split spoon sampling is generally used to collect undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted with a split spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augured hole and the core extracted.

When split spoon sampling is performed to gain geologic information, all work should



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be performed in accordance with ASTM D1586-98, "Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils".

The following procedures are used for collecting soil samples with a split spoon:

1. Assemble the sampler by aligning both sides of barrel and then screwing the drive shoe on the bottom and the head piece on top.
2. Place the sampler in a perpendicular position on the sample material.
3. Using a well ring, drive the tube. Do not drive past the bottom of the head piece or compression of the sample will result.
4. Record in the site logbook or on field data sheets the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth.
5. Withdraw the sampler, and open by unscrewing the bit and head and splitting the barrel. The amount of recovery and soil type should be recorded on the boring log. If a split sample is desired, a cleaned, stainless steel knife should be used to divide the tube contents in half, longitudinally. This sampler is typically available in 2 and 3 1/2 inch diameters. A larger barrel may be necessary to obtain the required sample volume.
6. Without disturbing the core, transfer it to appropriate labeled sample container(s) and seal tightly.

#### 7.2.5 Test Pit/Trench Excavation

A backhoe can be used to remove sections of soil, when detailed examination of soil characteristics are required. This is probably the most expensive sampling method because of the relatively high cost of backhoe operation.

The following procedures are used for collecting soil samples from test pits or trenches:

1. Prior to any excavation with a backhoe, it is important to ensure that all sampling locations are clear of overhead and buried utilities.
2. Review the site specific Health & Safety plan and ensure that all safety precautions including appropriate monitoring equipment are installed as required.





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3. Using the backhoe, excavate a trench approximately three feet wide and approximately one foot deep below the cleared sampling location. Place excavated soils on plastic sheets. Trenches greater than five feet deep must be sloped or protected by a shoring system, as required by OSHA regulations.
4. A shovel is used to remove a one to two inch layer of soil from the vertical face of the pit where sampling is to be done.
5. Samples are taken using a trowel, scoop, or coring device at the desired intervals. Be sure to scrape the vertical face at the point of sampling to remove any soil that may have fallen from above, and to expose fresh soil for sampling. In many instances, samples can be collected directly from the backhoe bucket.
6. If volatile organic analyses are required, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.
7. Abandon the pit or excavation according to applicable state regulations. Generally, shallow excavations can simply be backfilled with the removed soil material.

#### 8.0 CALCULATIONS

This section is not applicable to this SOP.

#### 9.0 QUALITY ASSURANCE/QUALITY CONTROL

There are no specific quality assurance (QA) activities which apply to the implementation of these procedures. However, the following QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.
2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration



# U. S. EPA ENVIRONMENTAL RESPONSE TEAM

## STANDARD OPERATING PROCEDURES

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DATE: 02/18/00

### SOIL SAMPLING

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activities must occur prior to sampling/operation, and they must be documented.

#### 10.0 DATA VALIDATION

This section is not applicable to this SOP.

#### 11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA and corporate health and safety procedures, in addition to the procedures specified in the site specific Health & Safety Plan..

#### 12.0 REFERENCES

Mason, B.J. 1983. Preparation of Soil Sampling Protocol: Technique and Strategies. EPA-600/4-83-020.

Barth, D.S. and B.J. Mason. 1984. Soil Sampling Quality Assurance User's Guide. EPA-600/4-84-043.

U.S. Environmental Protection Agency. 1984 Characterization of Hazardous Waste Sites - A Methods Manual: Volume II. Available Sampling Methods, Second Edition. EPA-600/4-84-076.

de Vera, E.R., B.P. Simmons, R.D. Stephen, and D.L. Storm. 1980. Samplers and Sampling Procedures for Hazardous Waste Streams. EPA-600/2-80-018.

ASTM D 1586-98, ASTM Committee on Standards, Philadelphia, PA.



# U. S. EPA ENVIRONMENTAL RESPONSE TEAM

## STANDARD OPERATING PROCEDURES

SOP: 2012  
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REV: 0.0  
DATE: 02/18/00

### SOIL SAMPLING

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APPENDIX A  
Figures  
SOP #2012  
February 2000



# U. S. EPA ENVIRONMENTAL RESPONSE TEAM

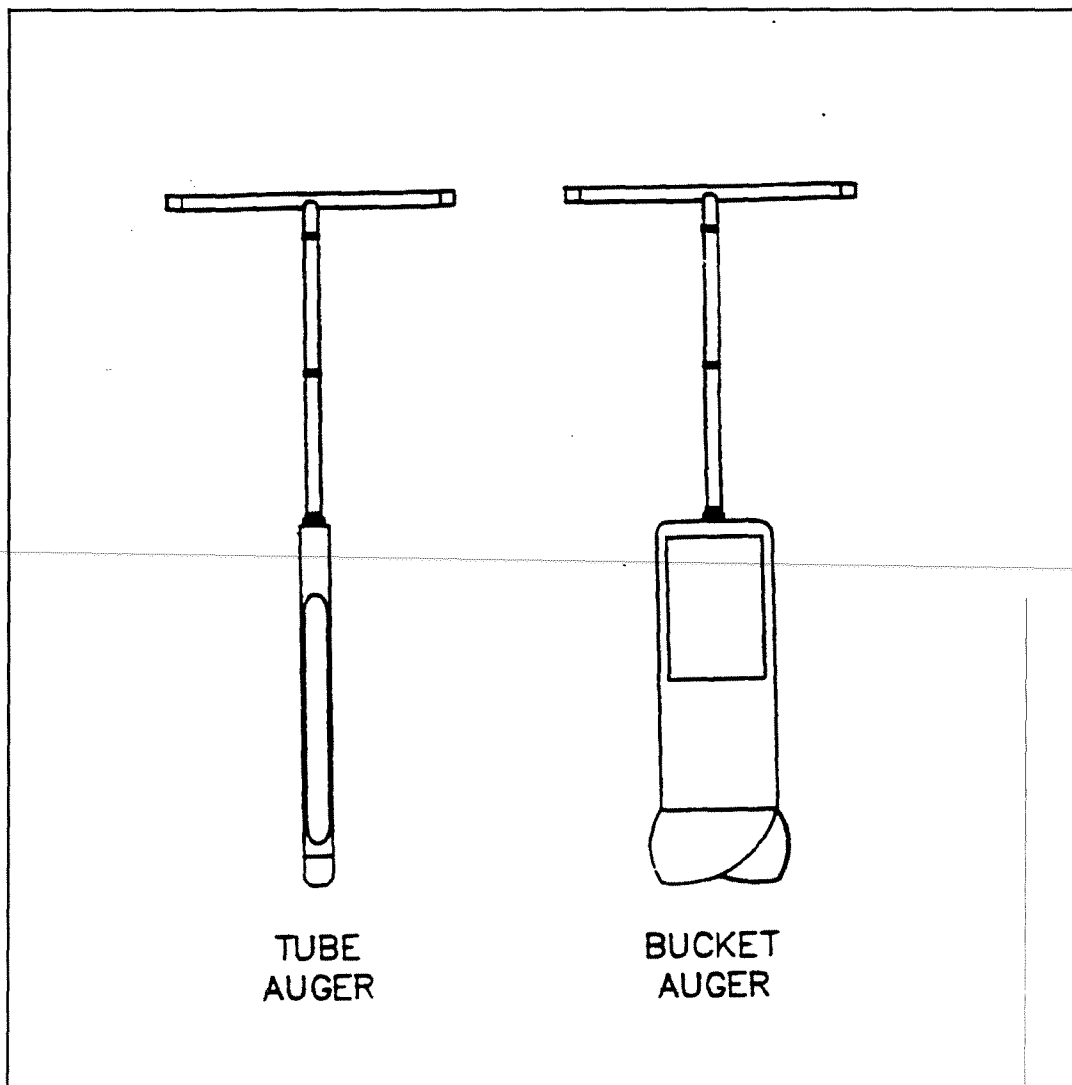
## STANDARD OPERATING PROCEDURES

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DATE: 02/18/00

### SOIL SAMPLING

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FIGURE 1. Sampling Augers





# U. S. EPA ENVIRONMENTAL RESPONSE TEAM

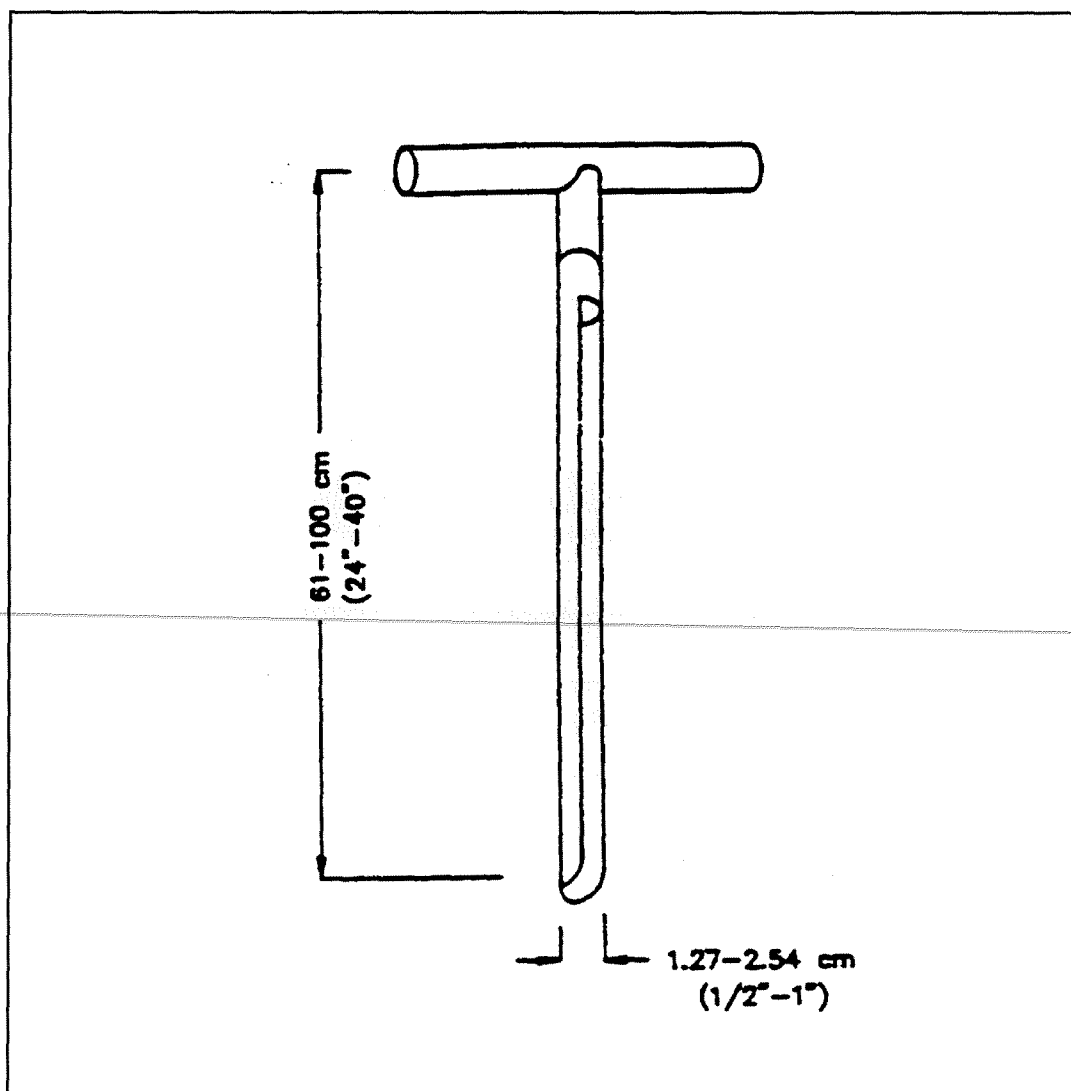
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### SOIL SAMPLING

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FIGURE 2. Sampling Trier







US EPA Region 7  
Standard Operating Procedure  
No. 4334.8A  
Surface Water Sampling  
November 2, 1982



STANDARD OPERATING PROCEDURE

No. 4230.17A

Surface Water Sample Collection

November 2, 1982

NOTE: This SOP is a pre-existing Environmental Monitoring & Compliance Branch SOP (SOP No. FW016A) which has been incorporated into the Environmental Services Division Operations & Quality Assurance Manual with a new SOP Number.

APPROVED:

John R. Melhi  
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Date

Charles P. Henry  
Regional Quality Assurance Officer

1/10/90  
Date

*certified 1/23/23*

STANDARD OPERATING PROCEDURE  
NO. FW016A  
SURFACE WATER SAMPLE COLLECTION

BY

Dale I. Bates

APPROVED:

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Coordinator Date

Robert B. Dona 11/2/02  
Section Chief Date

Bill Fairless 11/2/02  
Branch Chief Date

### 1. PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish uniform procedures for collecting water samples from surface waters.

### 2. APPLICATION

The procedures and guidelines provided in this SOP are applicable to all personnel of the Environmental Services Division who collect ambient water samples in support of Region VII programs.

### 3. GENERAL GUIDANCE

The overall objective of the specific study has to be established prior to conducting any surface water studies because the selection of sampling locations, type of samples to collect and parameters to analyze are all directly related to the study objectives. Some examples of objectives for surface water studies are:

1. Determine or evaluate the quality and/or trophic state of a water system.
2. Determine the effect of a specific discharge on a water body.
3. Determine the corrective measures needed to protect a water body or system for specific uses.
4. Obtain data on waste loads, water quality and characteristics that will permit prediction or modeling of the water system to describe probable water quality and effects on uses under a variety of conditions.
5. Trace the occurrence of specific constituents in a specific water system.

Other factors that may effect the selection of sampling locations and methodology include water use, point source discharges, non-point source discharges, tributaries, changes instream characteristics, types of stream beds, stream depth, turbulence, flow patterns, presence of structures (e.g., weirs, dams), accessibility and availability of personnel.

Due to the variability of objectives of a specific study and factors that may influence the conduct of the study, considerably more planning is required for ambient water quality studies.

### 4. SELECTION OF SAMPLING LOCATIONS

- a. Unless sampling locations have already been established, e.g., during a previous study, reconnaissance of the study area will have to be accomplished in order to locate suitable sampling sites.



b. A prime consideration in selecting sampling locations is accessibility. If the water body is navigable and a boat is to be utilized to collect samples, the sampling locations can be selected without regard to other means of access.

(1) Bridges are usually good choices as sites, because they provide ready access, are readily identifiable, and permit water sampling at any point across the width of the water body. However, it must be kept in mind that they may alter the flow pattern and may not always be located in desirable locations in reference to waste sources, tributaries, etc.

(2) On small streams and tributaries, the sampling locations may be selected on the basis of being accessible by wading. Personnel safety must be paramount when considering this method. Wading is not recommended in lakes, ponds and slow-moving rivers and streams, because bottom deposits are easily disturbed thereby resulting in the possibility of increased sediment in the overlying water column.

c. For ease of discussion, freshwater environments can be separated into two types: 1) rivers, streams and creeks, and 2) lakes, ponds and impoundments. These water bodies as a group exhibit different characteristics, therefore, sampling sites selection must be adopted to each.

(1) Rivers, streams and creeks. The areas that exhibit the greatest degree of cross-sectional homogeneity are the most suitable sampling locations. Since mixing is principally governed by turbulence and water velocity, the selection of a site immediately below a riffle area will insure good vertical mixing. Horizontal (cross-sectional) mixing occurs in constrictions in the channel. In the absence of turbulent areas, the selection of a site should be governed by the absence of tributaries and point source discharges.

(a) When several sampling stations along a stream reach are to be established, they should be spaced at intervals based on time of water travel, not distance. As a general rule of thumb, stations should be established at about one-half day time of travel intervals for the first two days of travel time below a waste source and approximately one day travel time intervals through the remaining distance. In very small or very turbulent streams, natural purification may be well advanced in as little as one to two days time of water travel and stations should be at much closer intervals than suggested above.

(b) If the study is to be compared with a previous study, the same sampling stations should be used, if possible, for comparison purposes.

(c) A station should be established wherever a marked physical change occurs in the stream channel. For example, if a stream reach consists of a long rapids section of swift shallow water with a rocky bottom followed by a long section of deep water section of slow-moving water with a muddy bottom, a sampling station should be established at both ends of the reach as well as between the rapids and quiet water sections to yield actual characteristics.

(d) Dams and weirs cause changes in physical characteristics of a stream that may be similar to the above rapids-quiet water situation, because they create quiet, deep pools that, by comparison were swift and shallow. Such impoundments should be bracketed by sampling stations. When time of water travels through them is long, stations should be established within the impoundments.

(e) Since stream structures, such as dams, provide significant reaeration of oxygen deficient water, stations should be established at short distances above and below the structures to measure the rapid, artificial increase in dissolved oxygen (D.O.) which is not representative of natural reaeration.

(f) A minimum of three sampling stations should be established between any two points of major change in a stream; e.g., a waste discharge, a tributary inflow, or significant difference in channel characteristics, even when the time of water travel between the points of change is short. The use of three stations is especially important when rates of change of unstable constituents are being determined.

(g) If the effects of a waste discharge or tributary on water quality are of interest, sampling stations should be established both upstream and downstream of the discharge or tributary. Unless a stream is extremely turbulent, it is difficult to measure the effect immediately downstream of the discharge or tributary. Since the inflow frequently hugs the stream bank with very little lateral mixing, care must be taken to establish the sampling station after complete mixing with the main stream.

(h) If tributary streams are important, there should be one station established near the mouth of the tributary and one on the main stream just above the confluence. The tributary sampling station should be established as near the mouth as feasible upstream of any mixing or effects from the main stream.

(i) Stations should be established as close as practicable to points or areas of important water uses.

(j) At stations where wastes and tributary waters are well-mixed, one sampling point near mid-channel is usually adequate. At stations where mixing is inadequate, the station should be sampled at quarter points across the width of the station.

(2) Lakes, ponds and impoundments. The location and number of sampling stations for these water bodies will vary according to the objectives of the study as well as the size and shape of the basin. The tendency to stratify due to temperature and density differences and the relative lack of mixing must be taken into consideration when establishing sampling stations.

(a) In ponds and small impoundments, a single station at the deepest point may be sufficient. In naturally-formed ponds, the deepest point is usually near the center; in impoundments, the deepest point is usually near the dam or spillway.

(b) In lakes and large impoundments, several sampling stations would be most representative and should be established utilizing a transect or grid.

(c) In lakes with irregular shapes and with several bays or coves that are protected from the wind, sampling stations should be established in these areas to adequately represent water quality.

(d) Sampling stations should be established where discharges, tributaries, land use characteristics, concentrations of population and other factors are suspected of influencing water quality.

(e) In order to have a basis of comparison of water quality, the establishment of control stations is usually necessary. A control station above a waste source is as important as are the stations below it and should be selected with equal care to ensure representative results. At times it may be desirable to locate two or three stations above a waste inflow to establish the rate at which unstable material is changing. The time of travel between the stations should be sufficient to permit accurate measurement of the change in the constituent under consideration.

## 5. TYPE OF SAMPLE

a. In most ambient water quality studies, grab samples will be collected. However, the objectives of the study will dictate.

b. For rivers, streams and creeks, the type of samples collected will be dependent upon the size of and amount of turbulence in the water body.

(1) With small streams less than 20 feet wide, a single grab sample collected at mid-depth in the center of the channel is usually adequate to represent the entire cross-section.

(2) For slightly larger streams, a vertical composite sample in the center of the channel would be most representative. The composite sample would consist of samples taken at just below the surface, at mid-depth and just above the bottom.

(3) For rivers, several vertical composite samples should be collected across the water body. The vertical composite samples should be collected at points in the cross-section approximately proportional to flow. The number of vertical composites required and the number of depths sampled for each are usually determined in the field. This determination is based on a reasonable balance between two considerations:

(a) The larger the number of subsamples, the more nearly the composite sample will represent the water body.

(b) Taking many subsamples is time-consuming and increase the chance of contamination.

(4) For lakes, ponds and impoundments, the greater tendency to stratify and the relative lack of adequate mixing usually requires that more subsamples be collected.

(a) In ponds and small impoundments, a single vertical composite sample at the deepest point is usually adequate.

(b) In lakes and larger impoundments, several vertical composites should be combined into a single sample. In some cases, it may be of interest to form several composites of the epilimnetic and hypolimnetic zones. Normally, however, a composite would consist of several verticals with subsamples collected at various depths.

## 6. PARAMETERS TO BE ANALYZED

a. The parameter to be analyzed will be dictated by the objective of the study.

b. When collecting vertical composites, the measurement of DO, pH, temperature, conductivity, etc., should be performed on each vertical aliquot.

## 7. METHOD OF SAMPLING

a. In general, any sampling method or sample collection device utilized to collect samples are acceptable provided they do not cause the integrity of the samples to be affected and they obtain samples that are representative of the water body being sampled.

b. The specific sampling method utilized will depend on the accessibility to, and the size and depth of the water body as well as the type of samples being collected.

(1) If surface water samples are being collected, direct dipping of the sample containers into the water is the most desirable collection method. This method may normally be utilized from a small boat, a low pier, etc., or by wading into the water body.

(2) If the water body is too deep or swift to wade or if samples must be collected from more than one water depth or from a bridge, etc., a sample collection device must be used.

c. For direct sample collection (i.e., surface water samples), the sample container should be placed in the water with the mouth facing upstream. The hand should be away from the mouth as far as possible.

d. Wading is an acceptable method if the stream or creek has a noticeable current (not impounded). Care should be taken to avoid biasing samples from bottom deposits rising when disturbed.

(1) The sampler should stand on the downstream side when collecting samples.

(2) When sampling a stream reach by wading, it is recommended that the sampling be accomplished starting from the downstream end to minimize the biasing of the samples from disturbance of bottom deposits.

e. There are several commercially manufactured sample collection devices available for collecting samples.

(1) For most samples, including priority pollutants, a teflon coated Nansen bottle or Dissolved Oxygen (D.O.) dunker.

(a) In addition to using the dunker for collecting samples for subsequent analyses, it has the capability for placing two DO bottles inside for collecting water directly into the bottles for DO determinations.

(b) A bacteriological or oil and grease bottle can also be attached to the top of the dunker for direct sample collection (surface water only).

(c) Except when the dunker's air vents are valved to allow opening at a specific depth, the dunker allows water to slowly enter the chamber as it moves downward through the water column. This allows a composite sample of the water column to be collected rather than a grab sample at a single depth.

(d) The Nansen bottle allows specific depth samples to be collected.

(2) If samples are desired at specific depths and the parameters to be analyzed don't require a teflon-coated sample collection device, a standard Kemmerer or VanDorn sampler may be used.

(a) The Kemmerer sampler is a brass cylinder with rubber stoppers that leave both ends open while being lowered through the water column in a vertical position.

(b) The VanDorn sampler is plastic and is lowered in the horizontal position.

(c) In both cases, a messenger is sent down the rope when the sampler is at the designated depth to cause the stoppers to close the cylinder. Water is removed through a valve to fill respective sample containers. With a rubber tube attached to the valve, DO sample bottles can be filled to overflowing.

(3) A bucket (plastic for most parameters except organics) may be utilized to collect surface water samples. The bucket should be rinsed twice with sample water prior to collection of the sample for analysis.

(4) Other suitable devices, either commercially manufactured or fabricated may be utilized provided the material of the device is compatible with the parameters to be analyzed.

f. When water sampling is performed in conjunction with sediment sampling at any sampling station, the water samples should always be collected first.



US EPA Region 7  
Standard Operating Procedure  
No. 4334.8.A  
Sediment Sampling  
January 13, 2000



US EPA Region 7 Superfund Division

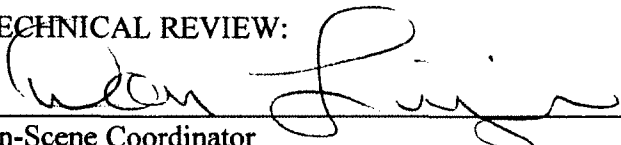
Standard Operating Procedure  
for  
Sediment Sample Collection

4334.8A

January 13, 2000

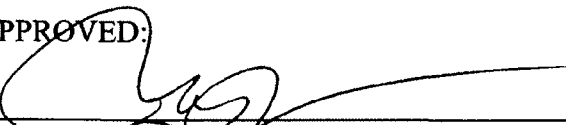
by Shirley L. Williams

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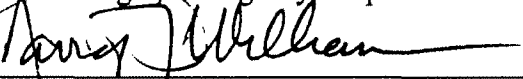
  
\_\_\_\_\_  
On-Scene Coordinator  
Emergency Response and Removal Branch

Date: 1/13/00

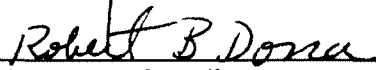
APPROVED:

  
\_\_\_\_\_  
Branch Manager, Emergency Response and Removal Branch

Date: 1-14-00

  
\_\_\_\_\_  
Branch Manager, Enforcement / Fund-Lead Removal Branch

Date: 1-18-00

  
\_\_\_\_\_  
Quality Assurance Coordinator  
Site Assessment and Cost Recovery Branch

Date: 1-20-00

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## 1.0 PURPOSE

The purpose of this Standard Operating Procedures (SOP) is to establish uniform procedures for the collection of bottom sediment samples from surface waters.

## 2.0 APPLICATION

The procedures contained herein are applicable to all personnel of the Superfund Division (SUPR) and their contractors who collect sediment samples in support of the Region 7 programs.

## 3.0 GENERAL GUIDELINES

The overall objectives of the specific sampling activity have to be established (in a site-specific Quality Assurance Project Plan [QAPP]) prior to initiation of sampling, because the selection of the sampling location, types of samples to collect, and parameters to be analyzed are all directly related to the study objectives. Factors that may affect the selection of sampling locations and the methodology include water uses, point source discharges, non-point source discharges, tributaries, changes in stream characteristics, types of stream beds, stream depth, turbulence, flow patterns, presence of structures (e.g., weirs, bridges, dams), accessibility and availability of personnel.

## 4.0 SELECTION OF SAMPLING LOCATIONS

1. Unless sampling locations have already been established; e.g., during previous sampling activities, reconnaissance of the area will have to be accomplished in order to establish suitable sampling sites.
2. A prime consideration in selecting sampling locations is accessibility.
  - a. Bridges and piers are good choices as they provide ready access.
3. Typical sediment deposition areas in rivers, streams and creeks are located inside river bends, downstream of islands and downstream of obstructions; e.g. bridge supports.
  - a. Constrictions in the channel should be avoided because the stream bottom may be scoured due to increased velocity.
  - b. Sites that are located immediately above or below the confluence of two streams or rivers should be avoided because flows from the two tributaries do not necessarily mix immediately and the sediment may be moving as two streams in proportion to the inflow from the two tributaries.
  - c. Potential sites upstream from the confluence with another stream may also be unsuitable at times due to possible backflow which can upset the normal movement of sediment.
  - d. A site immediately below a riffle area is good since the greatest deposition occurs where the stream velocity slows down.
  - e. In many circumstances, a number of sediment samples should be collected along a cross-section of a river or stream in order to adequately characterize the stream bed material. In such cases, samples should be collected at quarter points along the cross-section of the sampling site selected.



4. When collecting sediment samples in lakes, ponds and impoundments, the sampling site should be located approximately at the center of the water mass.

a. For reservoirs formed by the impoundment of rivers or streams, the coarser grained sediments are deposited near the headwaters of the reservoir and the bed sediments near the center of the water mass will be composed of fine-grained materials.

b. The shape, inflow pattern, depth and circulation should be considered when selecting sediment sampling sites in lakes and reservoirs.

## 5.0 TYPE OF SAMPLE

1. In most cases, a single grab sample of sediment will be collected at any sampling location.

2. When sampling requires that individual grab samples be combined into one sample; e.g., sampling at quarter points along the cross-section of a river, care must be taken to insure the individual grab samples are homogeneous and are of similar composition. Samples of dissimilar composition should not be combined but analyzed separately.

\*When sampling for volatile organics (VOAs), care must be taken to insure that the samples are sealed as quickly as possible with a minimum of handling so that the sample is representative. Please see EPA R7 ENSV SOP 2230.3B, "Sampling Soils for Determinations of Volatile Organic Compounds" for additional information.

## 6.0 METHODS OF SAMPLING

There are basically three methods of collecting sediment samples: dredging, coring, scooping (direct spooning).

### 6.1 DREDGING

This method of sampling involves the use of a clam shell type sampling collection device.

a. The Peterson dredge is preferable when the bottom is rocky, in very deep water and when the stream velocity is high. The dredge should be lowered very slowly as it approaches the bottom, because it can force out and miss lighter materials if allowed to drop freely.

b. The Eckman dredge performs well where bottom material is soft. It is unsuitable for hard bottoms and in streams with high flow velocities. It should also not be used from a bridge more than a few feet above the water, because the spring mechanism which activates the sampler can be damaged by the messenger if dropped from too great a height.

c. The Ponar dredge is one of the most effective samplers for general use. It is easy to use, prevents the escape of material by the addition of end plates and reduces the shock wave effect by the addition of a screen through the covering screen over the top of the sample compartment. Access to the secured sample through the covering screen permits sub sampling with coring tubes or Teflon scoops if contamination from metal parts is of concern.

## 6.2 CORING

This method of sampling involves the use of a tube to collect a vertical column of sediment. Coring devices are particularly useful in pollutant monitoring, because the shock wave effect is minimal; the sample is withdrawn intact permitting the removal of only those layers of interest; glass or Teflon core liners are available reducing the possibility of sample contamination; and the samples can be delivered to the laboratory for analysis in the tube in which they were collected. Due to relatively small surface area and sample size obtained, repetitive sampling may be required to obtain the required amount of sample.

a. These type of devices are recommended for sampling sediments for priority pollutant analyses.

b. In shallow, wadeable waters, the direct use of a core liner or tube (Teflon glass or variable diameter PVC) is recommended. Teflon or PVC is preferred to avoid glass breakage and possible sample loss and/or injury to sampling personnel. (PVC should not be used when collecting VOA samples.)

(1) Soft or semi-consolidated sediments such as muds and clays have a greater tendency to adhere to the inside of the tube, therefore, large diameter tubes can be utilized.

(2) Coarse or unconsolidated sediments such as sand and gravel tend to fall out of the tube, therefore, a small diameter tube should be utilized (2 inches in diameter is usually the best size).

(3) The tube should be about 12 inches in length and the wall thickness for Teflon, PVC or glass should be about 1/3 inch.

(4) Care should be exercised not to disturb the area to be sampled prior to collecting the actual sample.

(5) The core tube is pushed into the substrate until only 4 inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is pushed into the substrate will facilitate greater penetration and lessen core compaction. The tube is capped with a Teflon plug or sheet of Teflon held in place by a stopper or cork. After capping, the tube is slowly extracted. The bottom of the tube is also capped before removing it from the water.

c. For collecting sediment samples at depths that cannot be reached by hand, there are many types of coring devices that have been developed depending on the depth of water from which the sample is to be obtained, the nature of the bottom material and the length of the core to be collected.

## 6.3 SCOOPING

This method, as the name implies, involves the collection of the sample by scooping with a spoon or other such device.

a. If the water is wadeable, the easiest and best method to collect a sediment sample

is to scoop the sediment directly into the appropriate sample container. This method is accomplished by wading into the stream and, while facing upstream, by scooping up the sediment with a stainless steel or Teflon spoon from the stream bottom in the upstream direction. The sample is placed directly into the sample container.

b. If the stream has a significant flow and is too deep to wade, commercially manufactured samplers are available. However, it should be noted that this method may not be as effective as other devices, because the sampler may penetrate too deep in mud or other soft substrates and it is difficult to release secured samples in an undisturbed fashion in order to permit sub sampling.

## 7.0 REFERENCES

ERT SOP #2016, "Sediment Sampling", 11/17/94, Rev #: 0.0

EPA R7 ENSV SOP 2230.3B, "Sampling Soils for Determinations of Volatile Organic Compounds"

US EPA Region 7  
Standard Operating Procedure  
No. 4334.15A  
Groundwater Sampling  
January 13, 2000



US EPA Region 7 Superfund Division

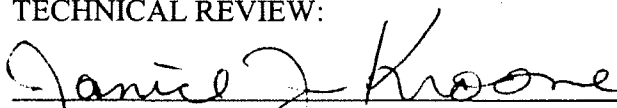
Standard Operating Procedure  
for  
Groundwater Sample Collection

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January 13, 2000

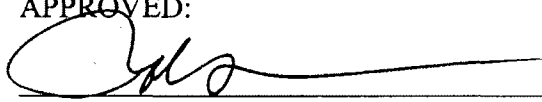
by Shirley L. Williams

TECHNICAL REVIEW:

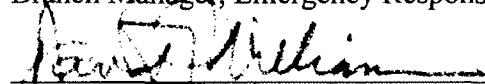
  
On-Scene Coordinator  
Emergency Response and Removal Branch

Date: 21 Feb 2000

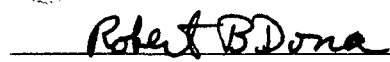
APPROVED:

  
Branch Manager, Emergency Response and Removal Branch

Date: 2-22-00

  
Branch Manager, Enforcement / Fund-Lead Removal Branch

Date: 2-22-2000

  
Superfund Quality Assurance Coordinator  
Site Assessment and Cost Recovery Branch

Date: 3-16-00



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## 1.0 PURPOSE

The purpose of this Standard Operating Procedures (SOP) is to establish uniform procedures for collecting representative samples of groundwater sources.

## 2.0 APPLICATION

The procedures outlined in this SOP are applicable to all personnel of the Superfund Division (SUPR) and their contractors involved in the collection of groundwater samples in support of Region 7 programs.

## 3.0 GENERAL CONSIDERATIONS

A. Since a comprehensive groundwater monitoring project may require the accumulation of extensive hydrogeologic information and consideration of a multitude of factors that affect subsurface water quality and quantity, the design of such a complex project is beyond the scope of this SOP. The development of a Quality Assurance Project Plan (QAPP) will have to address these factors on a case-by-case basis.

B. The procedures contained in this SOP will be restricted to the actual collection of samples and will not address in detail such topics as placement of wells, installation of wells, hydraulic gradient, subsurface and surface soil types and features, topography, land use, drainage patterns, etc.

## 4.0 SELECTION OF SAMPLING LOCATIONS

A. The site specific Addendum or QAPP for a specific activity or investigation should provide detailed information as to sampling locations.

1. As a general rule, a minimum of three wells are normally required to accurately assess subsurface conditions: one in the up gradient portion of the area of interest, one in the middle portion, and one in the down gradient portion. In some cases, however, a system of wells may be required to define the subsurface conditions, especially in establishing the depth to the shallow groundwater aquifer and the direction of the groundwater flow. Site conditions and the site specific Addendum or QAPP will determine the total number of wells required.

2. Existing wells should be utilized when possible and practicable to meet the needs of the project. The state Underground Storage Tank (UST) program should be contacted to see if any monitoring wells may exist that could provide valuable well placement information (e.e., geology, etc.) or data.

3. The installation of monitoring wells and specific siting of these wells should be addressed in the site specific quality assurance project plan.

B. Sampling location guidelines applicable to all groundwater sampling activities cannot feasibly be established since each sampling activity has unique characteristics based upon its hydrogeological setting. A systematic investigation of an area is normally required to determine the optimum

sampling locations. A review of existing information and investigation of surface and subsurface conditions are all correlated to determine the necessity, extent, location, and depth of groundwater sampling points required to meet the QAPP objectives.

C. When selecting sampling points the following factors should be considered:

1. Movement of groundwater from high to low gradient stagnant areas.
2. Groundwater movement induced by sampling and production wells.
3. Inflow-outflow from surface water.
4. The number and horizontal placement of wells should be governed by the hydrogeologic conditions.
5. The vertical placement of the sampling point should be governed by locating the withdrawal point at a representative depth to average concentration gradients or at a depth commensurate with the project objectives. The depth should be set so that seasonal fluctuations in the water table do not affect sample collection.

D. Small springs should be sampled in unconsolidated deposits by driving a well point or slotted pipe to a depth of 1 meter or less into the ground adjacent to the spring.

E. Large springs should be sampled in consolidated rock.

## 5.0 TYPE OF SAMPLES

A. Normally, only grab samples are manually collected of groundwater, because the movement of water is slow and the water quality does not normally exhibit sudden or drastic changes.

B. The site specific addendum or QAPP should identify the type of samples to collect.

C. The specific parameter to be analyzed may dictate that grab samples be collected.

## 6.0 PARAMETERS TO BE ANALYZED

A. The site specific addendum or QAPP should specify the parameters to be analyzed.

B. Some common parameters that indicate overall water quality and quantities of an area, but not specific pollution problems, for planning purposes are: pH, oxidation reduction potential, temperature, total dissolved solids, nitrate, chloride, water level, and Total Organic Carbon (TOC).

## 7.0 METHOD OF SAMPLING

A. Prior to any groundwater sampling, all wells should be adequately purged to eliminate all fine material from the area of the well screen, if applicable, and to clear the well of stagnant water.

1. As a general rule, the well should be purged until three to five times the volume of water in the well has been removed. As an alternate procedure, the well can be purged until the conductivity, pH, and temperature stabilize.

2. If a well is pumped dry during purging, it should be allowed to recharge before sampling.

3. There are various methods available for purging wells: Suction lift pumping (pitcher, centrifugal, roller, piston, or peristaltic pumps), pressure ejection pumping, submersible pumping, and bailing.

B. Samples may be collected by transporting the water to the surface in a container or transporting the water through a closed conduit and discharging at the surface.

1. A depth integrated or point sampler may be utilized to collect samples in a container.

- a. A depth integrated sampler is a container equipped with a holding and submerging mechanism which collects water throughout the vertical profile. Other depth integrating samplers known as bailers are lowered through the water and are filled through the bottom inlet which contains a check valve for retaining water when retrieved.

- b. A point sampler is used to collect a sample at a specific depth.

\* Special note. When collecting samples for volatile organic analysis (VOAs), care must be taken to assure the tubing used does not interfere with the analysis (i.e., add contamination or degrade analytes of concern)

2. In a closed conduit transport system, a pump, compressed gas or a vacuum is used to transport water to the surface.

- a. Vacuum systems should not be used for collecting volatile organic samples.

- b. Pressure or vacuum lysimeters may be utilized to collect samples from the vadose zone; i.e., the zone above the water table.

- c. Pressure or air lift piezometers installed in boring can also be used to collect samples in the vadose zone.

C. When sampling wells with in-place plumbing, samples should be collected following purging from a cold water tap as near to the well as possible, and before any treatment system (i.e., chlorination, fluoridation, water softener, etc.) if possible. Samples should be collected directly into the appropriate sample containers.

## 8.0 GROUNDWATER LEVEL MEASUREMENT

A. The measurement of the groundwater in the wells is conducted in conjunction with groundwater sampling. The data from such measurements is needed to determine the “free” water surface and can be used to establish groundwater gradients and ultimately, the direction of groundwater flow.

B. All groundwater level measurements should be made in reference to an established point (usually the top of the casing) on the well casing and be documented in the field records.

C. All measurements should be accomplished prior to purging the well.

D. Some specific techniques for measuring groundwater levels are as follows:

1. Popper or Bell Sounder. The bell or cup shaped weight that is hollow on the bottom is attached to a measuring tape and lowered into the well. A “plopping” or “popping” sound is heard when the weight strikes the surface of the water. A measurement is made by lifting and lowering the weight in short strokes and reading the tape when the weight barely strikes the water. The length of the weight must be subtracted from the reading if it is not included in the length of the tape.

2. Weighted Tape. This method is the same as (1) except any type of weight such as a padlock or lead sinker is fastened to the end of a tape.

3. Chalked Tape. The end (lower 2 to 5 ft) of a steel measuring tape is coated on either side with carpenter’s chalk, ordinary blackboard chalk, a dry (non-contaminated) soil, or paste that changes color when it gets wet. The end is weighted and lowered into the center of the well until a hollow “plopping” sound is heard when the weight strikes the water. The tape is lowered very slowly for at least another six inches, preferably to an even foot mark. The tape is carefully withdrawn from the well and the water depth is determined by subtracting the wetted length of tape plus the weight from the total measurement.

4. Electric Water Level Indicator or Sounder. This device consisting of a spool of small diameter cable with a probe attached to the end. A meter, light, and/or buzzer attached to the spool is activated when the sounder probe comes in contact with the water and completes the electrical circuit. The probe is usually required to be submerged to about 1 3/4 inches to fully activate the meter or other indicator.

5. Other Devices. There are commercially available water level indicators and recorders. These devices are primarily used for closed systems or permanent monitoring wells.

C. Whenever groundwater measurements are made, caution should be exercised to prevent inadvertent contamination of the well or cross-contamination between wells. The measuring device that comes in contact with the water should be adequately cleaned prior to and between uses.

## 9.0 INVESTIGATION DERIVED WASTEWATER

A. When sampling ground water, the Project Manager must ensure that contaminated water is handled in a manner that does not cause further contamination via runoff into a body of water,

sensitive area, or possibly into private drinking water wells.

B. It may be appropriate to collect the purge water and run it through a carbon filter, or other treatment mechanism, prior to discharge. A public owned treatment works (POTW) may be used to dispose of this water, or another method may be needed to ensure that the contaminated water is handled appropriately.





US EPA Region 7  
Standard Operating Procedure  
No. 2420.4D  
Field Chain of Custody for Environmental Samples  
January 2008



STANDARD OPERATING PROCEDURE

No. 2420.4D

FIELD CHAIN OF CUSTODY FOR ENVIRONMENTAL SAMPLES

January 8, 2008

by

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**Attachments**

1. RLAB Custody Seal;  
Total number of pages: 1.
2. Chain of Custody Record (COC);  
Total number of pages: 1.
3. Instructions for Completing a Chain of Custody Record (COC);  
Total number of pages: 3.

**A. Purpose**

The purpose of this Standard Operating Procedure (SOP) is to establish uniform policies and procedures for use by field personnel to maintain an accurate written record of environmental samples from the time of collection through their acceptance by a laboratory for analysis. The custody procedures utilized within the laboratory for receiving samples and maintaining custody through the analytical processes are not covered in this SOP. See "Storage and Security of Environmental Samples," SOP 2420.2 for custody procedures utilized within the Regional Laboratory (RLAB).

**B. Applicability**

The policies and procedures outlined in this SOP are applicable to all Environmental Services Division (ENSV) personnel, Environmental Protection Agency (EPA), state/local agencies, and/or EPA contractors who collect environmental field samples for analyses by the RLAB or contract laboratories.

**C. Summary of Procedures**

As a requirement of any activity which may be used to support litigation proceedings, the validity of any data introduced into evidence must be clearly demonstrated. In the case of samples collected in support of an enforcement case, it must be clearly documented that the sample introduced into evidence is, in fact, the same sample collected and/or that the analytical data offered into evidence accurately represent the environmental conditions at the time of sample collection. It is imperative that there is adequate proof to demonstrate that transfer, storage or analysis, and that the analytical results were obtained from the same sample collected. Therefore, an accurate written record must be maintained to track the possession and handling Chain of Custody Record (COC) (see Attachment 2) of each sample from the moment of collection through analysis and its introduction into evidence.

By definition, a sample is in "custody" if:

1. It is in one's actual physical possession; or
2. It is in one's view, after being in one's physical possession; or
3. It is locked up so no one can tamper with it, after being in one's physical possession; or
4. It is placed in a designated secured area



**D. Definitions/Acronyms**

ANOP	Analytical Operations Section
ANSETS	Analytical Services Tracking System
ASR	Analytical Services Request
CLP	Contract Laboratory Program
COC	Chain of Custody Record
ENSV	Environmental Services Division
EPA	Environmental Protection Agency
ESAT	Environmental Services Assistance Team
LIMS	Laboratory Information Management System
PM	Project Manager
PO	Project Officer
QC	Quality Control
REAP	Region 7 Environmental Analysis Program
RLAB	Regional Laboratory
RSCC	Regional Sample Control Coordinator
Sample Tags	Sample container labels
SOP	Standard Operating Procedure
SRN	Sample Receipt Notice
UPS	United Parcel Service
VOA	Volatiles

**E. Personnel Qualifications**

Personnel performing this task should have a basic knowledge of the RLAB sample and records management procedures.

**F. Responsibilities****1. Project Manager (PM)**

- a. The PM or designee (i.e., field contractor) submits a completed Analytical Services Request (ASR) to the RLAB 30 days before initiation of the sampling activity.
- b. The PM or designee (i.e., field contractor) ships and/or delivers properly collected, preserved, labeled, and packaged samples to the RLAB.

- c. The PM or designee (i.e., field contractor) is responsible for the accuracy and completeness of all accompanying paperwork. If any changes are required as a result of the sampling (e.g., sample number changes, additional analyses, samples not collected, quality control (QC) code additions), the PM or designee (i.e., field contractor) must see that these corrections are made on all paperwork.

All changes made to the paperwork (COC, sample tags (sample container label), or field sheets) must also be made to the information contained in the Laboratory Information Management System (LIMS). It is the responsibility of the PM or designee (i.e., field contractor) to supply correct information so that the Regional Sample Control Coordinator (RSCC) (or designated backup) can properly process the samples into the LIMS. Whenever possible, any changes are made prior to the delivery of the samples. If necessary, the RSCC (or designated backup) will assist the PM or designee (i.e., field contractor) when changes are noted prior to sample collection/delivery, concurrent to sample delivery or after.

- d. The PM must be available to help resolve any problems with the samples or must designate someone to do this for them in their absence. This requires that when delivering samples, the PM or designee (i.e., field contractor) stays with the RSCC (or designated backup) to answer any questions. Samples must not be just dropped off (unless after normal business hours).

The PM or designee (i.e., field contractor) calls the RSCC (or designated backup) close to the anticipated delivery date and/or time that samples are sent by courier (i.e., Federal Express) to confirm that samples have arrived and to answer any questions the RSCC (or designated backup) may have.

## 2. RSCC

- a. The RSCC (or designated backup) opens the ice chest (cooler) and utilizing the Infrared Digital Thermometer, checks the cooler temperature in three (3) different locations of the cooler (i.e., top, middle, and bottom of the cooler) and records the temperature range (in degrees Celsius) in the last row of the "Receiving Laboratory Remarks/Other Information" column on the COC (see Attachment 2) (i.e., cooler temperature received between 2-4 degrees Celsius).

- b. The RSCC (or designated backup) verifies the presence of all samples, checks all documentation and signs the COC after all paperwork is complete and accurate.
- c. The RSCC (or designated backup) works with the PM or designee (i.e., field contractor) to obtain correct information and puts the amended information into the LIMS.
- d. The RSCC (or designated backup) notifies the PM or designee (i.e., field contractor) of problems which prevent acceptance of the samples by ENSV. RLAB maintains all samples received in a secure location including those pending reconciliation of problems.
- e. The RSCC (or designated backup) logs samples into the LIMS (see "RLAB Procedures for Sample Receipt and Log-In", SOP No. 2420.1) and is responsible for the proper storage, tracking and/or distribution of the samples to the appropriate contract laboratories (this includes while the sample is in transit to the contract laboratory facility). The RSCC (or designated backup) prepares an electronic Sample Receipt Notice (SRN) message for each activity received by the RLAB and routes it to the Analytical Operations Section (ANOP) PM, LIMS Administrator, RSCC (or designated backup), and to the Environmental Services Assistance Team (ESAT) personnel for Analytical Services Tracking System (ANSETS) tracking.
- f. The RSCC (or designated backup) also electronically routes a SRN message to the appropriate RLAB staff as appropriately designated on the SRN, such as, ESAT, the Contract Laboratory Program (CLP) Project Officer (PO), or the Region 7 Environmental Analysis Program (REAP) PO, and/or appropriate back-up personnel and specific EPA analysts.

G. **Procedures**

- 1. In order to ensure adequate control and documentation of collected samples, the number of personnel handling the samples from the time of collection through delivery to RLAB should be limited.
- 2. The following actions must be accomplished in order to ensure that the relationship between the physical sample and the description of the sample is clearly, completely and accurately established, and that the custody of the sample is initiated from the time of actual sample collection.

- a. A unique number is assigned to each sample (see "Identification, Documentation, and Tracking of Samples," SOP No. 2420.5) in order to relate the descriptive information to a physical sample. If a sample consists of several containers for analysis of different parameters from the same physical sample, the same number is used for each portion of the original sample.
- b. A sample tag is securely attached to each container at the time of collection, for specific instructions for filling out the sample tag (see "Identification, Documentation and Tracking of Samples," SOP No. 2420.5).
- c. Custody of the sample is initiated at the time of collection by ensuring that the sample is in the sample collector's physical possession or view at all times, or is stored in a locked place where no one can tamper with it.

The sample collector is responsible for the collected samples until they are delivered to the RLAB.

3. Samples may be delivered to RLAB by the sampler or EPA contractor via courier or commercial carrier.
  - a. Sampler or EPA contractor-conveyed samples are those transported and delivered to RLAB. The coolers may be sealed or unsealed, but the sampler or EPA contractor must ensure that they are secured in the transport vehicle when he/she is not physically with the vehicle.
  - b. Samples may be delivered via courier (e.g., Greyhound). The cooler and sample containers must be transported with the lids secured. The transfer of possession of the samples must be recorded from the sampler or EPA contractor to RLAB.
  - c. Samples may be shipped via commercial carrier (e.g., Federal Express, Airborne, United Parcel Service (UPS)) from the field to RLAB. The cooler and sample containers must be sealed at the time of shipment.
4. Samples are considered to be sealed when they are packaged in such a manner that would prohibit tampering or readily reveal any tampering, if it occurred.

- a. A custody seal (see Attachment 1) may be used to secure the individual sample container, as appropriate to meet specific regulatory program requirements. These custody seals must be signed and dated by the sampler or EPA contractor when used to seal individual sample containers.
    - b. The use of a custody seal must be used to secure the openings of boxes, plastic bags, ice chests or coolers containing samples. These custody seals must be signed and dated by the sampler or EPA contractor when used to seal the shipping containers.
  5. The COC (see Attachment 2) is initiated at the time of sample collection and must accompany all samples. The COC is utilized to document the transfer of a sample from the sampler or EPA contractor through receipt by the RSCC (or designated backup) at RLAB.
- RLAB instructions for the completion of the COC are outlined in Attachment 3.
- a. The transfer of possession of the samples would occur when the sampler or EPA contractor delivers the samples to RLAB, gives them to the courier who will deliver the samples to RLAB, or packs the samples in a sealed shipping container for shipment to RLAB via commercial carrier.
    - b. The original and yellow copy of the COC will accompany the samples to RLAB. When the samples are conveyed by the sampler or EPA contractor, the COC may be hand carried. When the samples are delivered via courier or commercial carrier, the COC must be placed in a plastic document enclosure which is enclosed in the shipping container.
  6. When samples are delivered to RLAB after duty hours, the samples and the COC will be placed in the refrigerator located on the back dock until acceptance by the RSCC (or designated backup) in accordance with the procedures outlined in "Storage and Security of Environmental Samples," SOP No. 2420.2.
  7. Once RLAB has accepted the samples, the responsibility for custody of the samples transfers to the RLAB personnel. Custody of the samples is maintained through analysis in accordance with the laboratory's internal control procedures.
  8. The original of the completed COC is obtained by RLAB for inclusion with the permanent site activity data files, and is included with the final data transmittal sent to the PM.

9. The yellow copy of the completed COC is returned to the PM for inclusion in their appropriate activity files after all samples, for a given activity, have been accepted.
10. The yellow copy of the completed COC can be given to the person relinquishing the samples.
11. The custody seals or evidence tape associated with the specific samples or sample shipments are not retained.



#### H. **Quality Assurance/Quality Control**

A written tracking record (COC) is maintained from the time that the sample is collected to its transfer from the collection site to its laboratory destination. This record is used to demonstrate that sample possession has been secured and limited. Signed and dated custody seals placed over the access points of the sample shipment demonstrate that the contents of the samples have not been tampered with or compromised.

#### I. **References**

1. US EPA, Region 7, "RLAB Procedures for Sample Receipt and Log-In," Environmental Services Division Operations and Quality Assurance Manual, SOP 2420.1
2. US EPA, Region 7, "Identification, Documentation, and Tracking of Samples," Environmental Services Division Operations and Quality Assurance Manual, SOP 2420.5
3. US EPA, Region 7, "Storage and Security of Environmental Samples," Environmental Services Division Operations and Quality Assurance Manual, SOP 2420.2

Attachment 1  
RLAB Custody Seal

<b>CUSTODY SEAL</b>	_____	_____
	Date	Signature
		
		
<b>CUSTODY SEAL</b>	_____	_____
	Date	Signature



Attachment 2

CHAIN OF CUSTODY RECORD  
ENVIRONMENTAL PROTECTION AGENCY REGION VII

ACTIVITY LEADER(Print)		NAME OF SURVEY OR ACTIVITY		DATE OF COLLECTION		SHEET			
				DAY MONTH YEAR		of			
CONTENTS OF SHIPMENT									
SAMPLE NUMBER	TYPE OF CONTAINERS				SAMPLED MEDIA				RECEIVING LABORATORY REMARKS/OTHER INFORMATION (condition of samples upon receipt, other sample numbers, etc.)
	CUBITAINER	BOTTLE	BOTTLE	BOTTLE	VQA SET (2 VIALS EA)	WATER	SOIL	SEDIMENT	
	NUMBERS OF CONTAINERS PER SAMPLE NUMBER					ICE	SLURRY	OTHER	
DESCRIPTION OF SHIPMENT					MODE OF SHIPMENT				
_____ PIECE(S) CONSISTING OF _____ BOX(ES) _____ ICE CHEST(S); OTHER _____					_____ COMMERCIAL CARRIER: _____ _____ COURIER _____ SAMPLER CONVEYED _____ (SHIPPING DOCUMENT NUMBER)				
PERSONNEL CUSTODY RECORD									
RELINQUISHED BY (SAMPLER)		DATE	TIME	RECEIVED BY		REASON FOR CHANGE OF CUSTODY			
<input type="checkbox"/> SEALED <input type="checkbox"/> UNSEALED				<input type="checkbox"/> SEALED <input type="checkbox"/> UNSEALED					
RELINQUISHED BY		DATE	TIME	RECEIVED BY		REASON FOR CHANGE OF CUSTODY			
<input type="checkbox"/> SEALED <input type="checkbox"/> UNSEALED				<input type="checkbox"/> SEALED <input type="checkbox"/> UNSEALED					
RELINQUISHED BY		DATE	TIME	RECEIVED BY		REASON FOR CHANGE OF CUSTODY			
<input type="checkbox"/> SEALED <input type="checkbox"/> UNSEALED				<input type="checkbox"/> SEALED <input type="checkbox"/> UNSEALED					

**Attachment 3**  
**Instructions for Completing a Chain of Custody Record**

(Note: Each numbered item explains what is to be entered into that particular block moving from left to right, top to bottom of the document.)

1. Activity Leader. Enter the first initial and last name of the EPA Project Manager.
2. Name of Survey or Activity. Enter the activity number and/or Analytical Services Request (ASR) number (e.g., ERN07/900) for which the samples were collected.
3. Date of Collection. Enter the day, month, and year the samples were collected.
4. Sheet. Enter 1 of 1 unless there are more than one total sheets describing the shipment. If multiple sheets, enter the consecutive number of each sheet of the total number of sheets (e.g., 1 of 3, 2 of 3, 3 of 3).
5. Contents of the Shipment.
  - a. Enter the specific sample numbers, number of sample type containers per sample number and sample media in the appropriate column
    - (1) The ASR number and the individual sample numbers composing the shipment are entered in the "Sample Number" column (e.g., 2222-2). If more than one sheet is required, continue on additional sheets. For shipments of a large group of samples, it would be more appropriate and efficient to complete a separate sheet for each shipping container.
    - (2) The types of containers for each sample number are entered in the columns provided. The size should be entered above the container type, as appropriate. For Volatiles, the "VOA Set" refers to two=40 ml vials contained in the cubitainer which are collected for volatile organics analyses. The container types are modified, as necessary or appropriate, to describe sample containers.
    - (3) The sampled media for each sample number will be indicated by placing an "X" in the appropriate column. If the sample media is not listed, the actual media sampled should be entered in the "Other" column (e.g., wipe, sludge, air, biota, fish, etc.).
    - (4) The "Receiving Laboratory Remarks/Other Information" is to be used by the RLAB to indicate any problems with the shipment or condition of the

samples upon receipt; e.g., custody seal on sample container or shipping container broken, a sample container broken in transit, a sample lost due to leakage during shipment, etc. The temperature of the shipping coolers(s) are to be recorded in the lower area of this column. This column may also be used to record other sample numbers for cross-referencing purposes (e.g., external sample number).

- b. After entering all of the above information, the total contents of the shipment should be indicated by marking out any remaining lines in this section. This can be accomplished either by drawing a line across the next line after the last entry and entering "None to Follow" or "Activity/ASR Complete," or by drawing a line across the next blank line or diagonally across the remaining lines in the section and entering "None to Follow" or "Activity/ASR Complete."

6. Description of Shipment. Enter the total number of pieces (e.g., samples or sample containers) packed in the total number of shipping containers (e.g., ice chests, boxes or other, which comprise the total shipment) (e.g., 12 pieces in 2 ice chests or 24 pieces in 2 boxes).
7. Mode of Shipment. Indicate the mode by which the samples are shipped to the RLAB by placing an "X" in the appropriate line preceding the specific mode in this block. If the shipment is via commercial carrier, the name of the carrier and the shipping document number (e.g., airbill) should be entered in the appropriate lines provided. This information may be entered by the sample shipper (sampler or individual to whom the sampler relinquished the samples), or the shipment receiver (lab sample custodian), as appropriate.
8. Personnel Custody Record. This portion of the form provides the record of changes of custody of the shipment (sample or group of samples) from the sample collector to the laboratory. To provide an adequate written record, all of the blocks should be completed as described below.
  - a. The sample collector will sign the first "Relinquished By" block when the samples are presented to another individual or commercial carrier.
    - (1) An "X" should be entered in the appropriate block to indicate whether the shipment is sealed or unsealed with a piece of completed custody seal tape, the date and time when the samples are relinquished should be entered in the appropriate blocks, and the reason for change of custody (e.g., transport to lab, receipt by lab, etc.) should be entered in the appropriate block.

- (2) If the sampler is presenting the samples to a commercial carrier for shipment, the name of the carrier should be entered in the next available "Received By" block. The signature of a representative of the carrier is not required.
- b. Each individual who received the shipment of samples will sign the next available "Received By" block and enter an "X" in the appropriate block to indicate whether the samples were received sealed or unsealed with a piece of completed custody seal tape. If the samples were shipped via commercial carrier, the individual receiving the samples (e.g., sample custodian at the RLAB) should enter the date and time the samples were received and the reason for change of custody (e.g., receipt by the RLAB) in the appropriate blocks.
- c. Each successive individual who relinquishes custody of the samples will sign the next available "Relinquished By" block, enter an "X" in the appropriate block to indicate whether the sample shipment is sealed or unsealed with a piece of completed custody seal tape, enter the date and time when custody is relinquished and enter the reason for change of custody in the appropriate blocks.



US EPA Region 7  
Standard Operating Procedure  
No. 2420.5E  
Identification, Documentation, and Tracking of Samples  
August 28, 2007





# STANDARD OPERATING PROCEDURE

No. 2420.5E

## IDENTIFICATION, DOCUMENTATION, AND TRACKING OF SAMPLES

August 28, 2007

by

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**A. PURPOSE AND APPLICABILITY**

The purpose of this standard operating procedure (SOP) is to establish uniform procedures for assigning sample numbers, labeling sample containers, documenting the sample collection process, and for tracking samples.

The collection of samples is an essential step in the process for obtaining information on a variety of environmentally-related conditions and situations. Because the analytical results of samples are used extensively to support regulatory decisions, statutory actions, environmental and health assessments, and litigation proceedings, a critical component of the sample collection process is the proper identification, documentation, and tracking of each sample collected.

The procedures outlined herein are applicable to all samples received by the Region 7 Laboratory (RLAB) for analysis (either in-house analysis or out-source contract lab analysis) and to laboratory-generated quality control (QC) samples. The Regional Sample Control Coordinator (RSCC), or their designated backup, shall ensure, at the time of sample receipt, that samples received by RLAB conform to the identification and documentation requirements of this SOP. This SOP should be provided to all individuals (EPA, state, and tribal staff, plus their contractors) collecting samples for delivery to RLAB to facilitate compliance with these procedures.

**B. SUMMARY OF PROCEDURE**

1. Sample tracking is accomplished by using the Region 7 Laboratory Information Management System (LIMS). The LIMS is used to identify and track the status of all samples analyzed by the EPA Region 7 Laboratory and its contractors. The current LIMS is a product called R7LIMS. R7LIMS and any future LIMS products will follow the sample identification scheme defined in this SOP. Additionally, the LIMS can generate field sheets and tags (sample labels) to facilitate identification and documentation of field collected samples (see SOP 2420.13, "RLAB Procedures for Preparation of Field Sheets and Tags"). The physical location of samples is tracked by chain-of-custody procedures.
2. The identification and documentation of each sample is required in order to provide tangible evidence that shows the data resulting from sample analysis is linked directly to the sample collected. The basic mechanism used to establish this critical link between samples collected and analytical data is the assignment of a unique sample identifier to each sample collected, with supporting written information to document the sampling process. In addition to providing the means for establishing the relationship between samples and analytical results, the assignment of unique sample identifiers provides a means for tracking samples

through the analytical data generation process.

3. Sample identification is achieved by labeling each field collected sample with a unique sample identifier. Samples contained in multiple sample containers will bear the same unique sample identifier on each container, plus, each container will be uniquely identified (usually by analysis). Quality control is an integral part of the process of obtaining reliable information about environmental samples; therefore, field and laboratory quality control samples will be uniquely identified in an appropriate and consistent manner.
4. Sample documentation is accomplished by recording the appropriate information about the sample on a field sheet which bears the sample's unique sample identifier. If samples are delivered to RLAB with sample identifiers that are not consistent with the unique sample identifiers described in this SOP, the RSCC (or designated backup) will assign the requisite unique sample identifiers and record the original sample identifier, if any, in the LIMS "External Sample Number" field. Laboratory QC samples are documented on the sample prep and/or analysis log.
5. Because the identification and documentation of samples establishes the foundation for substantiating reported analytical data, it is important that the individuals who collect and/or generate samples follow the procedures contained in this SOP. The procedures contained in SOP No. 2420.4, "Field Chain-of-Custody for Environmental Samples," should be used in conjunction with this SOP to provide complete field sample documentation.

#### C. **DEFINITIONS**

The following definitions of commonly-used terms relating to types of samples and sampled matrices are provided for clarification in the sample identification process:

1. **Sample**. The word 'sample' is an often overworked term. It can refer to a sample collected in the field, a portion of a field sample that has been spiked with additional analytes (matrix spike sample), or a sample generated entirely within the laboratory, such as a method blank. The term 'sample' most often refers to a Field Sample that is of one matrix collected from a specific point (or area if spatially composited) at a specific time (or period of time if temporally composited). A sample may be divided into several different containers, each for a different type of analysis and possibly requiring different methods of preservation (see SOP 2420.6, "Sample Container Selection, Preservation and Holding Times"). It is common for all of these containers to be collectively referred to as being a (one) sample and for all of them to bear the same unique

sample identifier.

2. Field Sample. A representative portion of an environmental matrix (e.g. air, soil, water, etc.) collected from a specific location at a specific time to obtain information regarding environmental conditions and/or effects, process operations and material contents. Field Samples are actual portions of a matrix collected to determine its physical, chemical, or biological constituents and are distinguished from samples used for quality control (QC) purposes. Although QC samples collected in the field are in a sense field samples, the term Field Sample is used to denote a non-QC sample and is sometimes referred to as a “real” or “regular” Field Sample. Field Samples include those collected to evaluate background conditions and are categorized as grab, composite or continuous samples.
  - a. Grab Sample. A discrete portion of a matrix collected at a specific location at one instance in time (this period of time is typically defined as not exceeding 15 minutes to allow adequate time for sample collection under most field situations). This type of sample is representative of the environmental condition at the time of collection. This type of sample is commonly used for in-situ determinations and for obtaining information on constituents that require special handling or may be lost if sampled in another manner.
  - b. Composite Sample. A portion of a matrix consisting of a mixture of two or more discrete portions (grab samples) collected from a specific location over a period of time or from a specific area (multiple locations) at one time or over a period of time. This type of sample is a representative average of the environmental condition for a definable area and/or period of time. This type of sample is commonly used for assessing environmental conditions.
  - c. Continuous Sample. As the name implies, it is a representative portion of a matrix collected in an uninterrupted manner for a period of time. This type of sample is normally associated with in-situ determinations and is, therefore, not usually collected for submittal to a laboratory for analysis. Continuous samples are most commonly used for collecting data of air and water media; e.g., flow, pH, temperature, etc.
3. Split Sample. As the name implies, it is a sample that is separated or split from the total amount of material sampled and sent to a different laboratory for analysis. Soil matrix samples are homogenized then split to ensure uniformity. The Split samples are used to independently verify laboratory analysis.

4. Extract. An extract is the result of the extraction process. The sample extract is labeled by extraction personnel.
5. Digestate. A digestate is the result of the digestion process. The sample digestate is labeled by digestion personnel.
6. Quality Control Sample. Prepared in the laboratory, in the field, or combination thereof, a QC sample is incorporated into sample collection and/or analysis activities as a means of evaluating the quality of analytical results obtained from Field Samples. This type of sample may be a field-collected sample (e.g., duplicate sample) or a laboratory-generated sample, depending on its intended purpose, to evaluate and/or substantiate analytical results. Additional information on the use of QC samples for calculating data quality may be found in SOP No. 2410.15, "Estimating and Documenting Data Quality." The following types of QC samples are commonly encountered in sampling events and should be sufficient to categorize most QC samples:
  - a. Duplicate Sample. It is recognized that there are several interpretations of this term. For the purpose of calculating data quality, there are essentially two types of duplicate samples: field and laboratory, as described below.
    - (1) Field duplicate samples refer to two Field Samples collected simultaneously from the same location(s) under identical conditions. A duplicate grab sample consists of collecting two Field Samples at the same location and time. A duplicate composite sample consists of two Field Samples containing multiple grab samples each collected at the same location and time. If automatic samplers are used to collect composite samples, the collection of duplicate composite samples would require two automatic samplers to be collocated and set to collect the individual portions or aliquots at the same times. The dividing (also referred to as "splitting") of a single sample into two portions will be considered field duplicate samples in those situations where the preferred method of simultaneous collection cannot be met due to field conditions (e.g., the media being sampled is non-homogeneous like some soils, gravel, etc.).
    - (2) Laboratory duplicate samples refer to equivalent aliquots taken from a single sample received by a laboratory for analysis as unique samples. The process of obtaining the duplicate aliquots should be preceded by ensuring the sample is well mixed.

- b. Blank Sample. A sample that is presumed to be free of contamination from constituents of concern and is designed to detect contamination due to the sampling and/or analysis process (collection, preservation, handling, sampling environment, extraction, analysis, etc.).
- (1) Field Blank. Includes all blank samples which are prepared in or enter the field environment and include trip blanks, equipment blanks, bottle or container blanks, reagent or preservative blanks and tubing blanks. Ideally, a field blank for most analytical parameters should be exposed to the sampling, preservation and handling process used to collect the physical samples, but this may not always be possible (e.g., the field blanks for volatile organics are only transported unopened to and from the sampling environment). The type of field blank should be identified, as well as the group of Field Samples with which it is associated, in the appropriate sample documentation.
- (a) Trip Blank. It is a sample that is presumed to be free of contamination from constituents of concern, and is carried into the field and returned while being exposed to the same field conditions which the sample containers experience during the sample shipping process.
- (b) Tubing/Equipment Blank. It is a sample free from constituents of concern (normally deionized water that is distilled) and is pumped through or otherwise introduced into the sampling equipment. The process results in exposure of the sample to any constituents of concern which might be contained in or on the surfaces of the sampling equipment.
- (c) Preservation Reagent Blank. It is a sample which is originally free from constituents of concern (normally distilled deionized water) and to which the preservative (acid or other chemical) is added in the same concentration and quantity as normally added to a sample. The purpose is to determine if any contaminants of concern exist in the preservative used.
- (d) Container Blank. A sample originally free from constituents of concern (normally distilled deionized water) which is introduced into randomly chosen containers at the

time of sampling. The purpose of this blank is to determine the existence of contaminants of concern in the sampling containers.

- (2) Method Blank. A laboratory QC sample used to assess the level of contamination in the analytical system. A method blank is, typically, a portion of a clean matrix that is taken through the entire sample preparation and analysis process.
- c. Laboratory QC Sample. A variety of QC samples are used by an analytical laboratory for internal QC purposes. For the purpose of sample identification, all such samples prepared by the laboratory for internal use are classified under this category. Commonly used laboratory QC samples include lab duplicate samples, method blanks, lab control samples, reporting limit check samples, and matrix spikes.
- d. Performance Evaluation Sample. A sample that contains a known amount of a chemical constituent or parameter and is introduced for analysis to assess the accuracy of the analytical method. The actual content of the PE sample, either in regard to specific constituents and/or concentrations of constituents, is normally unknown to the receiving analytical laboratory.
- e. Proficiency Testing Sample. Similar to a performance evaluation sample except that it is provided by a NELAC (National Environmental Laboratory Accreditation Conference) certified PT sample provider. Results of the analysis of these samples are used for NELAC accreditation purposes.
- f. Some additional Field Samples may be thought of as QC samples due to the location or method of sample collection. These are labeled the same as, and analyzed the same as, other Field Samples.
  - (1) Rinsate Sample. This type of sample is used to evaluate the effectiveness of field decontamination procedures for sampling equipment. The sample is obtained by collecting the rinse water that is poured over the sampling equipment after decontamination has been completed (the water is normally distilled ionized water prepared in the laboratory and carried to the field).
  - (2) Background Sample. In some investigations, samples are collected to determine what is representative of the environment for constituents of concern. These samples, normally called



background samples, are Field Samples which are collected off-site or upstream of an area that is affected by a contaminant of concern, but are not expected to contain any or significant amounts of the contaminant of concern.

7. Matrix. The matrix (also known as ‘media’) refers to the substance from which the sample was obtained and/or of which the sample consists. Since the sampled matrix has a direct bearing on how a sample is preserved and on the selection of the method to analyze the sample, the identification of the matrix is an important aspect of sample documentation.
  - a. RLAB Matrix. The RLAB matrix is the matrix name used by RLAB to identify the matrix of the sample. It is the matrix used in the LIMS and in the RLAB Methods.
    - (1) Air. All samples collected to evaluate or analyze the chemical and physical contents of the air, both indoor and outdoor. The resulting sample may be in different forms depending on the method of collection (e.g., Tenex tube, canister, PUF, etc.).
    - (2) Solid. All samples obtained of soils, sediments, sludge, dust, and any other solid material.
    - (3) Tissue. All samples obtained of living organisms; e.g., plants or vegetation, fish, animals, etc., either whole or portions thereof.
    - (4) Waste. All samples obtained of media that do not logically fit under one of the other specifically defined matrices or contain exceedingly high concentrations of analytes. (Previously referred to as “Hazardous/Other.”) Examples of these type samples are wipe samples, drum samples, non-aqueous liquid samples, product or formulation samples and mixed media samples.
    - (5) Water. All samples obtained of aqueous liquid, e.g., wastewater, surface water, drinking water, groundwater, etc.
  - b. NELAC Matrix. NELAC has its own list of Quality System Matrices. These matrices are referenced in the RLAB Methods, but are not used in the LIMS or for sample definition/identification.
    - (1) Aqueous. Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes

surface water, groundwater, effluents, and TCLP or other extracts.

- (2) Drinking Water. Any aqueous sample that has been designated a potable or potential potable water source.
- (3) Saline/Estuarine. Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
- (4) Non-aqueous Liquid. Any organic liquid with <15% settleable solids.
- (5) Biological Tissue. Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- (6) Solids. Includes soils, sediments, sludges and other matrices with >15% settleable solids.
- (7) Chemical Waste. A product or by-product of an industrial process that results in a matrix not previously defined.
- (8) Air and Emissions. Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

#### D. **PERSONNEL QUALIFICATIONS**

Personnel collecting and/or delivering samples to RLAB should have a basic knowledge and understanding of RLAB sample management procedures including chain-of-custody (SOP 2420.4, "Field Chain-of-Custody for Environmental Samples"). RLAB personnel receiving samples must be knowledgeable of the sample log-in process (SOP 2420.1, "Sample Receipt and Log-in"). Personnel defining samples in the LIMS must be familiar with using the LIMS (SOP 2410.20, "R7LIMS Functions and Security") and have an R7LIMS account.

#### E. **SAMPLE IDENTIFICATION**

1. Each sample is identified by a unique sample identifier which is assigned to it.
  - a. This identifier is used to distinguish an individual sample from all other

samples and is used on all documentation relating to collection, handling, analysis and reporting the analytical results of an individual sample.

- b. Since a sample is normally analyzed for a number of different chemical constituents or parameters that require different sample containers and preservation techniques, the same unique sample identifier will be assigned to each portion of the original sample split among individual sample containers. For example, if a sample is split among three individual sample containers in order to properly preserve each portion for the specific parameter or group of parameters to be analyzed, each of the individual sample containers would be identified by the same unique sample identifier.
2. The unique sample identifier consists of three parts: the Analytical Services Request Number (ASR Number), Sample Number, and Quality Control Code (QC Code or QCC). These are frequently written together, separated by hyphens. The unique sample identifier is sometimes (confusingly) simply referred to as the sample number.
    - a. ASR Number - This is the number automatically assigned to an ASR at the time it is defined in the LIMS. Each ASR has its own unique number.
    - b. Sample Number - This number is assigned by the responsible Project Manager (or their designee) for each field sample collected for an ASR or by the analyst for each QC sample created for an ASR.
    - c. QC Code - This two or three character alpha code is used to identify the nature of the sample for QC purposes. Field personnel will normally only use the following codes to identify field collected samples:

__	=	Field Sample (two underscore characters)
FD	=	Field Duplicate
FB	=	Field Blank
FS	=	Field Spike
FSD	=	Field Spike Duplicate

Laboratory personnel will use the following codes to identify laboratory QC samples:

- |    |   |                      |
|----|---|----------------------|
| MB | = | Method Blank         |
| LD | = | Laboratory Duplicate |
| MS | = | Matrix Spike         |

MSD = Matrix Spike Duplicate  
LCS = Laboratory Control Sample  
RLC = Reporting Limit Check sample  
PE = Performance Evaluation sample  
PT = NELAC Proficiency Testing sample

3. The following examples are provided to illustrate some unique sample identifiers:

26-1-\_\_ - Field Sample number 1 for ASR Number 26  
26-1-FD - Field Duplicate of Field Sample above  
26-2-FB - Field Blank submitted for same ASR Number  
87-5-\_\_ - Field Sample number 5 for ASR Number 87  
87-5-MS - Matrix spike of Field Sample above  
87-900-LCS - Lab Control Sample number 900 for ASR Number 87

4. Some quality control samples have meaning only when referenced to another sample (i.e., QC Codes of FD, FS, FSD, LD, MS, MSD). To facilitate the identification of the referenced sample, the LIMS has two fields for use with these QC samples: Ref Sample Number and Ref QC Code. Rules for determining the Sample Number, Ref Sample Number, and Ref QC Code for these QC samples are given below.

- a. The QC sample and the referenced sample (the sample that the QC sample is a spike or duplicate of) must have the same ASR Number and Matrix.
- b. Field QC samples (FD, FS, FSD) will be assigned the same Sample Number as the original Field Sample (\_\_) that they are a duplicate or spike of. The Ref Sample Number, and Ref QC Code are automatically assigned by the LIMS and can not be edited by the user.
- c. Lab QC samples (LD, MS, MSD) that are a duplicate or spike of a Field Sample or Proficiency Testing sample (\_\_ , PT) will be assigned the same Sample Number as the original Field Sample or Proficiency Testing sample that they are a duplicate or spike of. By default, the Ref Sample Number will be set to the Sample Number and the Ref QC Code will be set to “\_\_” by the LIMS. If the sample being spiked or duplicated is a Proficiency Testing sample, a Ref QC Code of “PT” will need to be manually entered into the LIMS. Note that it is not appropriate for a Field Sample and a Proficiency Testing sample to have the same Sample Number.

- d. Lab QC samples (LD, MS, MSD) that are a duplicate or spike of any other field collected sample (QC Code of FB, FD, FS, FSD) must be assigned a different Sample Number than the original sample that they are a duplicate or spike of. The Ref Sample Number and Ref QC Code will need to be manually entered into the LIMS. Although not a requirement, it is suggested that a Sample Number in the “800” range be used for the lab QC sample.
  - e. MSD samples must have the same Sample Number, Ref Sample Number, and Ref QC Code as their associated MS sample. The MS sample must be defined in the LIMS before the MSD sample can be defined.
5. The following rules are provided for further clarification of the unique sample identifier assignment process:
- a. Each sample collected of a specific media will have a unique sample identifier. For example, if two samples are collected at the same location and time, but are of two different media (e.g., air and solid, or water and tissue), the sample of each specific media will be considered a separate sample. Each sample will be assigned a separate sample number.
  - b. In-situ samples collected for instantaneous field determinations (e.g., pH, temperature, specific conductance, dissolved oxygen, residual chlorine) in connection with the collection of samples for submission to a laboratory for analysis will be identified by sample identification numbers. Results of field determinations are recorded on field sheets associated with the sample collected for laboratory analysis. The sample identification number of the sample used for the field determination will normally be the same as the sample identification number of the sample submitted for analysis.
  - c. Continuous samples do not require the assignment of sample identification numbers, but do require specific written documentation to record sampling locations, and times of sampling and readings. Since many continuous monitors provide strip charts and/or printouts of readings, this documentation should be kept to supplement other written documentation.
  - d. Even though samples for some analyses, such as those for volatile organics, are always collected in two or more containers, they are considered to be a single sample. Additionally, if multiple analyses are to be performed (such as metals, pesticides and VOAs), separate containers will be needed for each analysis. These containers are collectively

considered to be one sample and will have the same unique sample identifier.

- e. Sample extracts are labeled by the person performing the extraction of the sample. The sample extract container is labeled by hand-copying the sample label's information onto a smaller sample extract label. The sample extract label must identify the extraction solvent. Transcription errors are prevented by double checking the sample extract label prior to affixing the sample extract label to the sample extract container. The sample extract label is then affixed to the sample extract container.
  - f. Digestates are labeled by the person performing the digestion of the sample. The sample digestate container is labeled by hand-copying the sample label's information onto a blank label. The sample digestate label must identify the requested analysis. Transcription errors are prevented by double checking the sample digestate label prior to affixing the sample digestate label to the sample digestate container. The sample digestate label is then affixed to the sample digestate container.
  - g. As a general rule-of-thumb, Field Blanks that are associated with a group of samples will have their own Sample Number. Field Blanks that are associated with just one Field Sample (e.g., a separate Field Blank for each Field Sample) may have, but are not required to have, the same Sample Number as the Field Sample that it is associated with.
  - h. It is common practice for some laboratory QC samples (MB, LCS, RLC) to be assigned a Sample Number in the "900" range. This is not a requirement for these samples (any number may be used), however, it is a desirable practice as it helps avoid confusion by keeping these QC samples "numerically segregated" from Field Samples. Additionally, the practice of numbering water QC samples beginning with 900 and soil QC samples beginning with 950 can also help minimize sample number "collisions." For sampling events involving a large number of Field Samples, running into the 900 range, it may be desirable to number these QC samples in the 1500, 2000, or other appropriate range.
6. All samples submitted for analysis will have a sample label affixed to each sample container.
- a. Sample labels currently in use are computer generated, therefore, minimal or no entries are required. Any entries made on the sample labels will be

accomplished using indelible ink.

- b. With the exception of volatile samples, samples packed inside a paint can for shipping and samples with extra volume for QC purposes, only one sample label is needed for each sample container. Since volatile, extra volume and over-packed samples consist of more than one container, multiple labels are required so that each container (including the outside container) can be labeled.

**Note:** Since some of the computer-generated sample labels (e.g. paper labels) are susceptible to deterioration from water, clear plastic tape should be placed over these sample labels if they will come into contact with water (including ice) during storage, transport and/or shipment. Some computer-generated sample labels (vinyl) are water resistant; these labels will not require tape protection.

- c. Each sample container must be uniquely identified by the sample label. Where there is only one container for an analysis (such as Metals in Water by ICP), the container is uniquely identified by the unique sample identifier (ASR Number, Sample Number, and QC Code) and the analysis abbreviation (such as “Met W.3B”). Where there is more than one container for an analysis (such as VOCs in Water by GC/MS), the containers are uniquely identified by the unique sample identifier (ASR Number, Sample Number, and QC Code), the analysis abbreviation (such as “VOA W.1E”), and a sequential container number (1, 2, 3, etc.). “Specific” sample container labels generated by the laboratory’s LIMS are uniquely identified as described above. When samples are received by the laboratory bearing LIMS “Generic” labels, labels generated by the sampler, or hand-made labels, the necessary additional information should be added to the label or a second label should be placed on the container to uniquely identify it. It is the responsibility of the laboratory person receiving the samples (RSCC or designated backup) to ensure that each container is uniquely identified.

## F. **SAMPLE DOCUMENTATION**

1. A field sheet is used to document the field sample collection process and contains pertinent information relative to the sample collected. (Laboratory QC samples are documented on the sample prep and/or analysis log as described in SOP 2410.10, “Analytical Data Submission Package Contents and Review.” This section deals primarily with field collected samples.)

2. A field sheet will be completed for each sample collected and will be the official document that provides a permanent record of each sample collected. Since this document is the essential written component required to establish the relationship between the sample collected and the analytical results obtained, it will be controlled and will become a part of the official file on a sampling event.
3. Field sheets can be generated by the laboratory's LIMS, or alternate forms may be used. A field sheet should contain, at a minimum, the following information:
  - a. Unique Sample Identifier - This may be recorded as three separate pieces of information (ASR Number, Sample Number and QC Code) or written as one entry (separated by hyphens).
  - b. Matrix Sampled - The RLAB matrix as defined in section C.7.a.
  - c. Project Information - This should include such things as the Project Manager, Project ID and description, city, state and other pertinent information.
  - d. Location/Description - This short description should identify, to the satisfaction of the Project Manager, where the sample was collected. This is typically done by describing or naming the sample collection location.
  - e. Sample Collection Date/Time - For time-composited samples, the start date and time and end date and time are required. For grab samples only the start date and time are needed. Times should be recorded in the 24-hour format.
  - f. Analyses - An unambiguous list of the required laboratory analyses.
  - g. Field Measurements - Recorded along with the measurement units.
  - h. Comments - As appropriate.
  - i. Sampler - The name of the person(s) collecting the sample.
4. The Project Manager is responsible for ensuring that all field sheets are properly and accurately completed, and are safeguarded until they are delivered to RLAB.
5. The original completed field sheets for each sampling activity will be delivered to RLAB along with the samples to be analyzed. They will be maintained in the RLAB analytical data file for the specific ASR.



6. All entries on the field sheets will be legible and completed in indelible ink. Corrections to entries on field sheets should be accomplished by drawing a single line through the entry to be corrected, entering the correction above or adjacent to the lined-through entry and dating and initialing the correction.
7. In addition to the field sheet, another essential component of sample documentation is chain-of-custody. SOP 2420.4, "Field Chain-of-Custody for Environmental Samples," describes the procedures for chain-of-custody of field collected samples being delivered to RLAB. SOP 2420.2, "Storage and Security of Environmental Samples," describes chain-of-custody procedures for within-lab sample transfers of routine samples. For samples that are connected with a criminal investigation, SOP 2420.10, "RLAB Procedures for Custody and Tracking of Samples and Analytical Data Files to be used as Evidence in Criminal Investigations," describes chain-of-custody documentation procedures for within-lab sample transfers.

G. **SAMPLE TRACKING**

1. The LIMS database system is used for tracking the status of samples and sample analyses through the analytical process and for tracking and reporting the results of sample analysis. Numerous reports are available from the LIMS and provide a variety of information pertaining to the samples and sample analyses. SOP 2410.20, "R7LIMS Functions and Security" and the LIMS online help provide more information on this.
2. Information relating to the status of samples submitted for analysis and the status of sample analyses may be obtained by the Project Manager from the LIMS or the RLAB Data Coordinator.
3. It is recognized that changes frequently occur in the field which result in changes to planned sampling activities. Since the LIMS system is used for logging in samples upon receipt, tracking, and ultimately reporting the results, it is essential that Project Managers ensure the entries contained in LIMS for specific sampling activities are accurate and complete (especially any field data and measurements). Discrepancies relating to numbers and types of samples and parameters requested for analysis must be corrected at the time of sample receipt by RLAB in accordance with SOP 2420.1, "Sample Receipt and Log-in".
4. SOP 2420.2, "Storage and Security of Environmental Samples" describes, for routine samples, the procedures for tracking the location of samples and sample containers within the laboratory. For samples that are connected with a criminal

investigation, SOP 2420.10, “RLAB Procedures for Custody and Tracking of Samples and Analytical Data Files to be used as Evidence in Criminal Investigations” describes the procedures used for tracking the location of samples and sample containers within the laboratory.

5. Unless otherwise requested, environmental samples will be properly disposed of in accordance with SOP 2420.9, “Sample Disposal,” upon completion of the analysis and finalization of the analytical results.

#### H. **QUALITY ASSURANCE AND QUALITY CONTROL**

It is incumbent on all parties involved with sample collection, analysis, and management that these procedures be followed. Conformance with these procedures shall be evaluated during scheduled audits of RLAB operations as described in SOP 2430.5, “Quality Control Spot Checks of Regional Laboratory Data Packages,” and SOP 2430.6, “Periodic Internal Program Review of the Region 7 Laboratory.”

#### I. **REFERENCES**

1. Region 7 SOP 2410.10, Analytical Data Submission Package Contents and Review
2. Region 7 SOP 2410.15, Estimating and Documenting Data Quality
3. Region 7 SOP 2410.20, R7LIMS Functions and Security
4. Region 7 SOP 2420.1, Sample Receipt and Log-in
5. Region 7 SOP 2420.2, Storage and Security of Environmental Samples
6. Region 7 SOP 2420.4, Field Chain-of-Custody for Environmental Samples
7. Region 7 SOP 2420.6, Sample Container Selection, Preservation and Holding Times
8. Region 7 SOP 2420.9, Sample Disposal
9. Region 7 SOP 2420.10, RLAB Procedures for Custody and Tracking of Samples and Analytical Data Files to be used as Evidence in Criminal Investigations

10. Region 7 SOP 2420.13, RLAB Procedures for Preparation of Field Sheets and Tags
11. Region 7 SOP 2430.5, Quality Control Spot Checks of Regional Laboratory Data Packages
12. Region 7 SOP 2430.6, Periodic Internal Program Review of the Region 7 Laboratory



US EPA Region 7  
Standard Operating Procedure  
No. 2420.6E  
Sample Container, Selection, Preservation, and Holding Times  
March 14, 2006



STANDARD OPERATING PROCEDURE

No. 2420.6E

SAMPLE CONTAINER SELECTION, PRESERVATION AND HOLDING TIMES

March 14, 2006

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**Attachments**

1. Guide for Sample Container Selection, Sample Preservation, and Holding Times  
Total number of pages: 5.
2. Guide for Selecting Intermediate Sample Container Material  
Total number of pages: 3.



**A. PURPOSE AND APPLICABILITY**

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for the selection of the proper sample containers and intermediate sample collection containers or devices when collecting samples for specific constituents (parameters) or groups of constituents; and for determining sample preservation and the holding times of samples from the time of collection until analysis is performed.

The guidance contained herein is applicable to all personnel who collect environmental samples for analysis by the Environmental Services Division (ENSV), including EPA and contractor personnel.

**B. DEFINITIONS**

ASR	Analytical Services Request
BNA	Base-Neutral/Acid Extractable
BOD	Biochemical Oxygen Demand
CBOD	Carbonaceous Biochemical Oxygen Demand
CFR	Code of Federal Regulations
DBCP	1,2-Dibromo-3-chloropropane
DO	Dissolved Oxygen
EDB	Ethylene Dibromide
ENSV	Environmental Services Division
GFF	Glass Fiber Filter
HEM	Hexane Extractable Material
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NPDES	National Pollutant Discharge Elimination System
PCB	Polychlorinated Biphenyl
PE	Performance Evaluation
PM	Project Manager
QC	Quality Control
RLAB	Regional Laboratory Branch
RSTC	Regional Science and Technology Center (facility where the RLAB is located)
RSCC	Regional Sample Control Coordinator
SOP	Standard Operating Procedure
SSR	Sampling Supplies and QC/PE Samples Request
SW	Solid Waste
TCLP	Toxicity Characteristic Leachate Procedure
TKN	Total Kjeldahl Nitrogen

TOX	Total Organic Halogens
TPH	Total Petroleum Hydrocarbons

C. **SAMPLE CONTAINERS**

1. The use of the proper sample container is extremely important to ensure the representativeness of the analytical data obtained, the sufficiency of the sample volume for analysis, and the non-interference or contamination of the sample resulting from the sample container material. When considering the sufficiency of the sample volume for analysis, any matrix spike and matrix spike duplicates (MS/MSD) must be accounted for, and the sampling volume should be adjusted as necessary (One sample for each analyte in each sampling event should at least be double the normal volume or larger volumes for all samples could be used. For example, since water samples for extractables are being collected in 1-gallon bottles, there should be sufficient volume to perform MS/MSD analysis on any sample in the batch.)
2. Special care will be taken to avoid any inadvertent contamination of sample containers prior to or during the sample collection process. Specifically:
  - a. Sample containers shall be left sealed or containerized during storage and transport to the sampling location and until the time of actual sample collection. [Exception: Polyethylene cubitainers are received from the manufacturer with the screw caps not attached. The cubitainers are collapsed and nested. The caps are screwed on to the cubitainers after they are filled with sample.]
  - b. Sample containers will not be rinsed with the media being sampled during the sample collection process unless specifically required for a given parameter or sampling process (e.g., collecting water samples for toxicity testing).

**Note:** The specific SOPs on sample collection should be referred to for the appropriate collection procedure to be used.
3. The sample container selection process is governed by:
  - a. The parameter or group of parameters to be analyzed; this includes the desired level of detection in many cases.
  - b. The media or matrix to be sampled (i.e., air, solid, tissue, water or other).

- c. The analytical method to be used for the analysis.
  - d. The laboratory (i.e., EPA Region 7 laboratory or a contract laboratory) to perform the analysis. The laboratory where samples will be analyzed is determined by the Laboratory Branch (RLAB) upon receipt of an Analytical Services Request (ASR) form.
- 4. Attachment 1 provides specific guidance for use in Region 7 in selecting the proper sample container by parameter, or parameter group, and the media being sampled. Additional guidance or requirements for acceptable materials of sample containers are contained, by the parameter to be analyzed, in the current 40 CFR Parts 136 and 141, and SW-846. Also, guidelines are normally found in the specific analytical methods and sampling procedures.
  - 5. When the use of intermediate sample collection containers is necessary, guidance on recommended intermediate container materials may be found in Attachment 2.

**D. SAMPLE PRESERVATION**

- 1. The immediate on-site analysis of samples at the time of collection is, in most cases, neither possible nor practical. Therefore, methods have been established to maintain the integrity of the sample until analysis can be accomplished. Even when samples are preserved in an appropriate manner, they should be analyzed as soon as possible after collection. An integral part of preservation is the selection of the proper sample container, the pretreatment of a sample container (if necessary), and the holding time allowable prior to analysis.
- 2. The purpose of sample preservation is to 1) retard biological action; 2) retard hydrolysis of chemical compounds and complexes; 3) reduce volatility of constituents; and 4) reduce absorption effects. The preservation methods used are generally limited to pH control, chemical addition, refrigeration or freezing. As a rule, the refrigeration (or icing) of samples should be utilized to maintain the samples at a temperature of  $4 \pm 2^{\circ}\text{C}$  during sample collection (including the collection of time or flow-weighted composite samples), transport, and storage.
- 3. The current guidance for sample preservation for use in Region 7 is provided in Attachment 1. Although taken into consideration when preparing this guidance, additional specific guidelines and requirements for sample preservation may be found in regulations (e.g., 40 CFR Part 136), publications (e.g., SW-846) and applicable analytical methods.

4. The following guidance is provided for field personnel to use when preserving the types of samples indicated:
  - a. Grab Samples: The applicable preservation method must be accomplished immediately upon sample collection.
  - b. Manually Composited Water Samples: The applicable preservation must be added, in full, to the initial aliquot and thus be available for each subsequent aliquot.
  - c. Automatically Composited Water Samples:
    - (1) When collected for either a single parameter or a parameter group where the type and amount of preservative required are identical, the applicable preservative is added to each container receiving an aliquot, prior to compositing.
    - (2) When collecting a composite sample that will later be split to create samples for a variety of individual parameters and each of these parameters requires different preservation methods, the samples collected for the composite should be iced to maintain a temperature of 4 °C until the compositing and splitting can be completed. The appropriate preservative is then added at the time the composite is split into separate containers.
5. Samples of the following media will not be preserved with the addition of any chemical compound, but will be chilled to 4 °C after collection and during transport and storage.
  - a. Solids: soil, sediment, sludge
  - b. Tissue (or freeze, -15 to -20 °C)
  - c. Other: non-aqueous solutions, product samples (liquid or solid), drum samples, wipe samples
6. The following parameters require special procedures:
  - a. **Biochemical Oxygen Demand (BOD)/Carbonaceous Biochemical Oxygen Demand (CBOD).** Water samples of chlorinated effluents collected for analysis of this parameter must be labeled with the word "CHLORINATED" on the sample tag to alert laboratory personnel.

Chlorinated samples require different analytical procedures than unchlorinated samples for this parameter.

- b. **Cyanide, Total and Amenable to Chlorination.** Water samples for these parameters should not be collected using automatic samplers, but should be collected manually either as a grab or a composite of several grab samples which are preserved at the time of collection. Since oxidizing agents such as chlorine decompose many cyanides, the sample must be treated to eliminate such agents, if they are present, at the time of collection. The presence of chlorine is determined by testing a drop of the sample with potassium iodide (KI)-starch test paper. A change in the color of the paper to blue indicates the need for treating the sample with a dechlorination agent. This treatment is accomplished by adding ascorbic acid, a few crystals at a time followed by the subsequent testing of a drop of sample until no color is produced on the KI indicator paper. An additional 0.6 gram of ascorbic acid is then added for each liter of sample volume. Preservation of the sample is then accomplished by adding 2 mL of 10 N sodium hydroxide solution or 10 pellets of sodium hydroxide crystals per liter of sample (to pH  $\geq$  12) and by icing the sample to 4°C during transport and storage.
- c. **Dissolved Oxygen (DO).** Water samples for this parameter are collected only on a grab basis. When collecting a sample for this parameter, the sample bottle should be filled to overflowing to ensure that no air bubbles are entrapped in the bottle when the stopper is replaced. When immediate measurement is not possible on site utilizing the DO probe method, the sample will be “fixed” immediately upon collection by first adding 2 ml of manganous sulfate ( $\text{MnSO}_4$ ) solution and then 2 ml of alkali-iodide-azide solution well below the surface of the liquid. The sample is mixed by inverting the bottle several times while holding the stopper in place and allowing to set until the floc has settled half way. Carefully remove the stopper and immediately add 2 ml of concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$ ) by allowing the acid to run down the neck of the bottle. Re-stopper and mix again and store at 10-20°C out of direct sunlight. Completion of the analysis for DO utilizing the Winkler titration method (Azide Modification) should be accomplished as soon as possible after collection and fixing, but not more than 8 hours after collection.
- d. **Metals, Dissolved.** Water samples for this parameter must be filtered on site utilizing a 0.45 $\mu\text{m}$  membrane filter as soon as practical after collection and then acidified with 5 mL 1:1  $\text{HNO}_3$  per liter of sample.

- e. **Microbiology (Total and Fecal Coliform; Fecal Streptococci).** Water samples for these parameters will be collected only on a grab basis. For chlorinated effluents, sodium thiosulfate (0.008%  $\text{Na}_2\text{S}_2\text{O}_3$ ) is added to the sample. This dechlorinating agent is normally added during sample container preparation and is, therefore, normally present in the sample container. Care must be taken during sample collection to avoid overfilling or rinsing out the agent. In addition, care must be taken to avoid contamination of the sterile sample container prior to or during sample collection; i.e., leave cap on container until ready to collect sample and do not place fingers in container or on the inside of the cap, while collecting a sample. An air space should be left at the top of the container after the sample is collected. Ice to 4°C.
- f. **Oil and Grease (Hexane Extractable Material, HEM)** Water samples for this parameter will be collected in one liter glass bottles on a grab basis only and acidified with 1:1 HCl to pH < 2 immediately after collection. The sample container should never be rinsed with the water or wastewater because these constituents tend to adhere to the sides of the container. Care should be taken to avoid contamination of the sample from fingers placed in the container or on the inside liner of the cap. In addition, enough air space should be allowed in the container to allow for the addition of the preservative.
- g. **Organics, Volatiles.** Grab samples only are collected for these parameters. Each sample will consist of two (2) 40-mL vials. Generally, four (4) 40-mL vials per water sample will be collected for low detection level and drinking water samples.
  - (1) Drinking water samples containing residual chlorine must be treated with sodium thiosulfate or ascorbic acid (depending on the analytical method) at the time of collection.
  - (2) Wastewater samples containing residual chlorine must be treated with ascorbic acid (25 mg per 40 mL) at the time of collection. These dechlorinating agents must be placed in the vials prior to collecting the samples.
  - (3) When collecting water samples, fill the sample bottles to overflowing, but take care not to flush out the sodium thiosulfate or ascorbic acid, if present. No air bubbles should pass through the sample as the bottle is filled, or be trapped in the sample when the bottle is sealed. After collection, the pH of the sample is adjusted

to a pH < 2 by carefully adding one drop of 1:1 HCl for each 20 mL of sample volume. Seal the sample bottles teflon-face down, and shake vigorously for 1 minute. A proper seal can be checked by inverting the sample and lightly tapping the end on a solid surface. If air bubbles are present, open the vial, add additional sample, reseal, and recheck for air bubbles. Store samples out of direct sunlight.

- h. **Phenols (Phenolics).** Water samples for this parameter should not be collected by using automatic samplers, but should be collected as a grab or a manual composite of grabs and preserved with 2 mL H<sub>2</sub>SO<sub>4</sub> to a pH < 2 at the time of collection. The samples should be iced to maintain them at 4°C during transport and storage.

#### E. SAMPLE HOLDING TIMES

1. The issue of holding times for samples is critical in the sample collection and analysis process, because the integrity of the samples can be affected depending on the parameter to be analyzed. Sample holding times are defined as the period of time between sample collection and initiation of sample analysis. In the case of timed composite samples, the holding time starts at the end of the compositing period (i.e., at the time the last portion of the composite sample is obtained).
2. Since holding times can affect the validity of the reported analytical results (especially in certain media programs and in enforcement actions), everyone involved in planning and executing sampling activities; planning and performing analyses; and reviewing analytical results must be cognizant of the implications of exceeding them during the process. In many instances, the holding times are required by specific regulations (e.g., 40 CFR Part 136 for wastewater samples under the NPDES program), while many others are recommended. Also, see Footnote 8 of Attachment 1.
3. Although many of the holding times contained in Attachment 1 were derived from regulatory requirements, the holding times should be considered as guidelines. When making decisions on the validity of analytical results based on holding times, personnel should consult appropriate regulations to determine if there are specific requirements for sample holding times.

#### F. OBTAINING SAMPLING SUPPLIES

1. When sampling supplies (e.g., sample containers, sample collection devices, preservatives, etc.) are needed, the project manager for the specific sampling

activity requests the necessary supplies on the Sampling Supplies and QC/PE Samples Request (SSR).

2. The requestor can pick up the supplies at the ENSV warehouse facility (3150 Dodge). The preservatives must be picked up at the RSTC (300 Minnesota Avenue). It is recommended that the requestor contact the Regional Sample Control Coordinator (RSCC) or designated back-up at the RLAB before going to either facility to ensure the supplies are ready for issuance.

G. **REFERENCES**

1. Code of Federal Regulations, Title 40 (40 CFR), Part 136.
2. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), U.S. EPA.
3. Standard Methods for the Examination of Water and Wastewater, Joint Editorial Board, American Public Health Association, American Water Works Association and Water Environment Federation, Latest Edition.



Attachment 1**Guide for Sample Container Selection, Preservation and Holding Times**

Parameter <sup>1</sup>	Sample Container <sup>2</sup>	Preservation	Holding Time <sup>8</sup>
<b>I. <u>Air and Gaseous</u></b>			
Dioxins/Furans	Puff glass jar	Cool 4°C	365 days
Metals on High-Vol GFF	1-gal. resealable plastic bag	Ambient Temperature	
Ozone Precursors			
Ambient levels	6-L canister	None	60 days
Source levels	400 mL canister	None	60 days
Particulate Matter on High-Vol GFF	1-gal. resealable plastic bag	Ambient Temperature	
Pesticides/PCBs	Puff glass jar	Cool 4°C	7 days
Semivolatiles/BNA	Puff/XAD glass jar	Cool 4°C	14 days
Volatile Organics			
Ambient levels	6-L canister	None	60 days
Source levels	400 mL canister	None	60 days
GC/MS Scan	6-L canister	None	30 days
<b>II. <u>Soil, Sediment and Solids</u></b>			
<b>A. Non-Product Samples</b>			
Asbestos	Plastic jar	None	None
Cyanide	8-oz. glass jar	Cool 4°C	28 days
Dioxin/Furans	8-oz. glass jar/sealable plastic bag in 1-qt. paint can	Cool 4°C	365 days
Explosives	8-oz. glass jar	Cool 4°C	14 days
Flashpoint	8-oz. glass jar	Cool 4°C	
GC/MS Scan, SemiVOA	8-oz. glass jar	Cool 4°C, store in dark	14 days
GC/MS Scan, VOA	2, 40mL VOA vials	Cool 4°C	14 days
Herbicides	8-oz. glass jar	Cool 4°C	14 days
Metals			
All metals except Cr <sup>+6</sup> and Hg, collected separately	8-oz. glass jar	Cool 4°C	180 days
Mercury	8-oz. glass jar	Cool 4°C	180 days
Cr <sup>+6</sup>	8-oz. glass jar	Cool 4°C	30 days
Methanol	8-oz. glass jar	Cool 4°C	14 days
Nutrients			
Nitrogen (NH <sub>3</sub> , NO <sub>2</sub> , TKN)	8-oz. glass jar	Cool 4°C	28 days
Phosphorous, total	8-oz. glass jar	Cool 4°C	
Oil and Grease	8-oz. glass jar	Cool 4°C	
pH	8-oz. glass jar	Cool 4°C	None
Phenolics (colorimetric)	8-oz. glass jar	Cool 4°C	28 days
Perchlorate	8-oz. glass jar	Cool 4°C	28 days
Pesticides/PCBs	8-oz. glass jar	Cool 4°C	14 days
Radioactivity	8-oz. glass jar	Cool 4°C	180 days

Parameter	Sample Container	Preservative	Holding Time
<b>II. Soil, Sediment, and Solids (cont.)</b>			
<b>A. Non-Product Samples (cont.)</b>			
Semivolatiles/BNA	8-oz. glass jar	Cool 4°C	14 days
Sulfate/Sulfide	8-oz. glass jar	Cool 4°C	28 days
Soil Toxicity Test	1-gal. ziplock bag	Cool 4°C	56 days
Total Organic Carbon	8-oz. glass jar	Cool 4°C	28 days
Total Petroleum Hydrocarbons (TPH)	8-oz. glass jar	Cool 4°C	14 days
SemiVOA			
VOA	2, 40-mL glass vials	Cool 4°C	14 days
Total Kjeldahl Nitrogen	8-oz. glass jar	Cool 4°C	None
Organic Parameters All except volatile organics	8-oz. glass jar	Cool 4°C	14 days
Volatile organics <sup>3</sup>	2, 40-mL glass vials	Cool 4°C or MeOH & Cool 4°C or NaHSO <sub>4</sub> & Cool 4°C	14 days
TCLP Metals, except Hg	8-oz. glass jar	Cool 4°C	180 days to extract, 180 days after extraction (required)
Mercury	8-oz. glass jar	Cool 4°C	28 days to extract, 28 days after extraction (required)
Volatile organics	2, 40-mL glass vials	Cool 4°C	14 days to extract, 14 days after extraction (required)
Semivolatile organics	8-oz. glass jar	Cool 4°C	14 days to extract, 7 days to extraction, 40 days after extraction (required)
Pesticides/Herbicides	8-oz. glass jar	Cool 4°C	14 days
<b>B. Product Samples</b>			
All parameters	8-oz. glass jar/sealable plastic bag in paint can	Cool 4°C	
<b>III. Tissue</b>			
Fish, collected for whole body/edible portion, all parameters	Double wrapped in heavy duty foil	Freeze	
Resectioned tissue, collected for:			180 days
Metals	Double wrapped in heavy duty foil	Freeze	
Semivolatiles	Double wrapped in heavy duty foil	Freeze	
Volatiles	Double wrapped in heavy duty foil	Freeze	
Foliage			
Herbicides/Pesticides	Double wrapped in heavy duty aluminum foil	Freeze	
Macroinvertebrates <sup>6</sup>		70% ethanol	6 months
Periphyton <sup>6</sup>			
Chlorophyll A		Freeze, store in dark	30 days
Enumeration		5% formalin, Cool 4°C, store in dark	6 months
Dioxins/Furans	Double wrapped in heavy duty foil	Freeze	365 days
Phytoplankton, collected for <sup>6</sup>			
Chlorophyll A		Cool 4°C, store in dark	14 days
Enumeration		5% formalin	6 months

Parameter	Sample Container	Preservative	Holding Time
<b>IV. Aqueous Samples</b>			
Chlorine Dioxide	1-L plastic cubitainer	Cool 4°C	None
Chlorophyll A	4-L plastic cubitainer	Cool 4°C, store in dark	14 days
Coliform, fecal	300-mL sterile plastic bottle	Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	6 hours
Dioxins/Furans	1-L amber glass bottle	Cool 4°C	365 days
Dissolved Organic Carbon	1-L amber glass bottle	H <sub>2</sub> SO <sub>4</sub> to pH<2 Cool 4°C	28 days
Explosives	128-oz. amber glass bottle	Cool 4°C	7 days to extract, 40 days after extraction
Herbicides	128-oz. amber glass bottle	Cool 4°C	7 days to extract, 40 days after extraction
Flashpoint	8-oz. glass jar	Cool 4°C	
Metals (except Hg and Cr <sup>+6</sup> )			
Total and acid soluble	1-L plastic cubitainer	HNO <sub>3</sub> to pH<2	6 months
Dissolved	1-L plastic cubitainer	Filter HNO <sub>3</sub> to pH<2	6 months
Chromium, hexavalent	1-L plastic cubitainer	Cool 4°C	24 hours
Mercury	1-L plastic cubitainer	HNO <sub>3</sub> to pH<2	28 days
Strontium	1-L plastic cubitainer	HNO <sub>3</sub> to pH<2	6 months
Acid, %	1-L plastic cubitainer	Cool 4°C	None
Alkalinity	1-L plastic cubitainer	Cool 4°C	14 days
BOD/CBOD	1-L plastic cubitainer	Cool 4°C	48 hours
COD	1-L plastic cubitainer	H <sub>2</sub> SO <sub>4</sub> to pH<2 Cool 4°C	28 days
Chlorine (residual)	1-L plastic cubitainer	Cool 4°C	1 day
Conductivity	1-L plastic cubitainer	Cool 4°C	28 days
Cyanide (total and amenable To chlorine)	1-L plastic cubitainer	(Ascorbic acid), NaOH to pH>12, Cool 4°C	14 days
Halides (Br <sup>-</sup> , Cl <sup>-</sup> , F <sup>-</sup> )	1-L plastic cubitainer	Cool 4°C	28 days
Haloacetic Acids/Dalapon	1-L amber glass bottle	Ammonium Chloride, Cool 4°C	14 days
Hardness	1-L plastic cubitainer	HNO <sub>3</sub> to pH<2, Cool 4°C	6 months
Inorganic Anions	1-L plastic cubitainer	EDA, Cool 4°C	14 days
Methane, Ethane, Ethene	2, 40-mL VOA vials	Cool 4°C	7 days
Methanol	1-L amber glass bottle	Cool 4°C	7 days
Nonfilterable Solids (NFS)	1-L plastic cubitainer	Cool 4°C	7 days
Oxygen, Dissolved (Winkler)	300-mL glass BOD bottle with Attached ground glass stopper	MnSO <sub>4</sub> + Alkali- Iodide-Azide, H <sub>2</sub> SO <sub>4</sub>	8 hours
Dissolved Oxygen, probe Method	1-L plastic cubitainer	Cool 4°C	1 day

Parameter	Sample Container	Preservative	Holding Time
<b>IV. <u>Aqueous Samples (cont.)</u></b>			
pH	1-L plastic cubitainer	Cool 4°C	determine immediately
Perchlorate	1-L plastic cubitainer	Cool 4°C	28 days
Residue			
All but settleable	1-L plastic cubitainer	Cool 4°C	7 days
Settleable	1-L plastic cubitainer	Cool 4°C	48 hours
Sulfate	1-L plastic cubitainer	Cool 4°C	28 days
Sulfide	1-L plastic cubitainer	Zinc acetate + NaOH to pH>9, Cool 4°C	7 days
Total Dissolved Solids (TDS)	1-L plastic cubitainer	Cool 4°C	7 days
Total Kjeldahl Nitrogen	1-L plastic cubitainer	H <sub>2</sub> SO <sub>4</sub> to pH<2.5, Cool 4°C	28 days
Total Solids	1-L plastic cubitainer	Cool 4°C	7 days
Turbidity	1-L plastic cubitainer	Cool 4°C	48 hours
Nutrients			
Nitrogen-Ammonia	1-L plastic cubitainer	H <sub>2</sub> SO <sub>4</sub> to pH<2, Cool 4°C	28 days
Nitrogen-Organic	1-L plastic cubitainer	H <sub>2</sub> SO <sub>4</sub> to pH<2, Cool 4°C	28 days
Nitrate	1-L plastic cubitainer	Cool 4°C	48 hours
Nitrate-Nitrite	1-L plastic cubitainer	H <sub>2</sub> SO <sub>4</sub> to pH<2, Cool 4°C	28 days
Nitrite	1-L plastic cubitainer	Cool 4°C	48 hours
Phosphorous (total)	1-L plastic cubitainer	H <sub>2</sub> SO <sub>4</sub> to pH<2.5, Cool 4°C	28 days
Ortho-phosphate	4-oz. plastic bottle	Cool 4°C	48 hours
Phosphorous, (dissolved)	1-L plastic cubitainer	Filter, H <sub>2</sub> SO <sub>4</sub> to pH<2, Cool 4°C	28 days
Carbamates <sup>10</sup>	60-mL screw cap vial	1.8 mL monochloroacetic acid buffer pH<3, Cool 4°C	14 days
Oil and Grease (HEM)	1-L glass jar	1:1 HCL to pH<2, Cool 4°C	28 days
Pesticides/PCBs	128-oz. amber glass bottle	Cool 4°C	7 days to extract, 40 days after extraction <sup>9</sup>
Phenolics	1-L glass jar	H <sub>2</sub> SO <sub>4</sub> to pH<2, Cool 4°C	28 days
Radionuclides	1-L plastic cubitainer	HNO <sub>3</sub> to pH<2	6 months (water:alpha/beta) 5 days(gamma; DW:alpha/beta)
Semivolatile/BNA	128-oz. amber glass bottle	Cool 4°C, store in dark	7 days to extract, 40 days after extraction
GC/MS Scan (BNA)	128-oz. amber glass bottle	Cool 4°C, store in dark	7 days
Total Organic Carbon (TOC)	1-L amber glass bottle	H <sub>2</sub> SO <sub>4</sub> to pH<2, Cool 4°C	28 days
Total Organic Halogens (TOX)	8-oz. amber glass bottle	Cool 4°C	7 days

Parameter	Sample Container	Preservative	Holding Time
<b>IV. Aqueous Samples (cont.)</b>			
Total Petroleum Hydrocarbons (TPH)			
SemiVOA	128-oz. amber glass bottle	Cool 4°C	7 days
VOA	2, 40-mL VOA vials	Cool 4°C	14 days
Toxicity Tests			
Acute	10-L (2.5-gal) plastic cubitainer	Cool 4°C	36 hours
Bioscreen (24 or 48 hr.)	4-L (1-gal.) plastic cubitainer or glass bottle	Cool 4°C	24 hours
Chronic	20-L (2.5/5-gal.) plastic cubitainer	Cool 4°C	36 hours
Triazine Herbicides	128-oz. amber glass bottle	Cool 4°C	14 days
Tritium	8-oz. glass jar	None	5 days
Volatile Organics <sup>3, 5</sup>			
Purgeable halocarbons	2, 40-mL glass vials	HCl to pH<2, Cool 4°C	14 days
Purgeable aromatic hydrocarbons	128-oz. amber glass bottle	HCl to pH<2, Cool 4°C	7 days
Routine Detection Level	2, 40-mL glass vials	HCl to pH<2, Cool 4°C	14 days
Low Detection Level	4, 40-mL glass vials	HCl to pH<2, Cool 4°C	14 days
EDB/DBCP	2, 40-mL glass vials	Sodium Thiosulfate Cool 4°C	14 days
GC/MS Scan (VOA)	2, 40-mL VOA vials	HCl to pH<2, Cool 4°C	14 days
<b>V. Liquid, Non-Aqueous</b>			
TCLP ( > 5% solids) <sup>4</sup>			
Mercury	8-oz. glass jar	Cool 4°C	28 days to TCLP extract, 28 days after extraction
Metals, except Hg	8-oz. glass bottle	Cool 4°C	180 days
Volatile Organics	2, 40-mL glass vials	Cool 4°C	14 days to TCLP extract, 14 days after extraction
Semivolatile Organics	8-oz. glass jar	Cool 4°C	14 days to TCLP extract, 7 days to extraction, 40 days after extraction
Pesticides/Herbicides	128-oz. amber glass bottle	Cool 4°C	7 days
Organic Parameters			
All except volatile organics	8-oz glass jar	Cool 4°C	7 days to extract, 40 days after extraction
Volatile organics	8-oz glass jar	Cool 4°C	14 days
<b>VI. Wipe Samples<sup>7</sup></b>			
Arsenic	8-oz. glass jar	Cool 4°C	
Cyanide	8-oz. glass jar	Cool 4°C	
Dioxin	8-oz. glass jar	Cool 4°C	365 days
Herbicides/Pesticides	8-oz. glass jar	Cool 4°C	14 days
Metals	8-oz. glass jar	Cool 4°C	180 days
Picric Acid	8-oz. glass jar	Cool 4°C	
Organic other parameters			
All except volatile organics	8-oz. glass jar	Cool 4°C	7 days to extract, 40 days after extraction
Volatile organics	8-oz. glass jar	Cool 4°C	

Parameter	Sample Container	Preservative	Holding Time
<b>VI. Wipe Samples (cont.)</b>			
TCLP (> 0.5% solids) Metals, except mercury	8-oz. glass jar	Cool 4°C	180 days to TCLP extract, 180 days after extraction

<sup>1</sup> Non-product and product sample definitions: A non-product sample is a sample which consists primarily of naturally occurring materials that may contain mechanically or chemically manufactured materials or substances as contaminants. A product sample is a sample which is known to consist primarily of a mechanically or chemically manufactured material that does not otherwise occur naturally in the immediate environment being sampled. A sample which cannot be identified as a non-product sample should be considered a product sample.

<sup>2</sup> All glass containers require a Teflon-lined lid or cap.

<sup>3</sup> These parameters are always collected as replicates. The sample container consists of two or four 40-mL glass vials (VOA vials) and an activated carbon filled thimble contained in a 1-L plastic cubitainer.

<sup>4</sup> § 40 CFR Part 261 Appendix II, 2.1: For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8  $\mu$ m glass fiber filter, is defined as the TCLP extract.

<sup>5</sup> Sodium Thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ) or Ascorbic Acid ( $\text{C}_6\text{H}_8\text{O}_6$ ) is utilized to de-chlorinate samples of chlorinated water or wastewater prior to pH adjustment. For drinking water samples, consult the applicable method to determine appropriate dechlorinating agent.

<sup>6</sup> Sample containers, preservation, and holding times vary. The information provided is Regional guidance. For compliance samples, consult appropriate references for complete preservation requirements.

<sup>7</sup> Each jar should contain a medical gauze pad.

<sup>8</sup> Generally, there are no required holding limits for air, soil, product and wipe media. (Exceptions: see Chapter 4 of SW-846). However, it is recommended that samples be analyzed within the holding time limits established in aqueous media for the specific analytes or analyte groups.

<sup>9</sup> Method 505 holding time 14 days (7 days for Heptachlor), preserve with Sodium Thiosulfate, 4°C, container 40-mL vial.

<sup>10</sup> Method 531.1 samples must be preserved to a pH<3 using monochloroacetic acid to minimize degradation of oxamyl, 3-hydroxycarbofuran, aldicarb sulfoxide, and carbaryl in neutral and basic waters. If residual chlorine is present add 80 mg of sodium thiosulfate per liter of sample to the sample bottle prior to collecting the sample.

Attachment 2**Guide for Selecting Intermediate Sample Container Material**

Media Sampled/Parameter	Intermediate Sample Container Material
<b>I. <u>Soil, Sediment and Solids</u></b>	
<b>A. Non-Product and Product Samples</b>	
All parameters except volatile organics	Glass, Aluminum
Volatile Organics <sup>1</sup>	Glass, Stainless Steel, Aluminum
<b>II. <u>Tissue</u></b>	
<b>A. Fish</b>	
All parameters	Glass, Plastic
<b>III. <u>Liquids</u></b>	
<b>A. Aqueous Samples</b>	
Chlorophyll A	Artificial substrate (glass slide)
Coliform, fecal <sup>2</sup>	Glass or Plastic
Dioxin/Furans	Glass, Stainless Steel (solvent rinsed)
Explosives	Glass
<b>Metals:</b>	
All except Cr <sup>6</sup>	Glass, Plastic, Automatic sampler equipped with Tygon intake tubing and glass or plastic compositing container.
Chromium, hexavalent	Glass, Plastic
<b>Minerals and Dissolved Materials:</b>	
Acid (%), Alkalinity, BOD, Chloride, Conductivity, Hardness, Residue, Sulfate, Turbidity	Glass, Plastic, Stainless steel, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container
Chlorine	Glass, Plastic, Stainless steel
COD	Glass, Plastic, Stainless steel, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container
Cyanide	Glass, Plastic, Stainless steel

Media Sampled/Parameter	Intermediate Sample Container Material
<b>III. Liquids (cont.)</b>	
<b>A. Aqueous Samples (cont.)</b>	
Fluoride	Plastic, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and plastic compositing container
Oxygen, dissolved	Glass, Plastic, Stainless steel
pH, lab or field	Glass, Plastic, Stainless steel, Teflon
Sulfide	Glass, Plastic, Stainless steel
Nutrients (N & P)	Glass, Plastic, Stainless steel, Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container
Oil and Grease <sup>2</sup>	Glass
Pesticides/PCBs	Glass, Stainless steel, Automatic sampler equipped with Teflon intake tubing and glass compositing container (cleaned and solvent rinsed)
Phenols/Phenolics	Glass, Plastic, Stainless steel
Radionuclides	Glass, Plastic
Semivolatiles/BNA	Glass, Stainless steel, Automatic sampler equipped with Teflon intake tubing and glass compositing container (cleaned and solvent rinsed)
Total Organic Carbon	Automatic sampler equipped with Tygon (or Teflon) intake tubing, and glass or plastic compositing container
Total Organic Halogens	Glass, Plastic
Toxicity Tests: Acute Bioscreen Chronic	Automatic sampler equipped with Teflon or new Tygon tubing, and glass or Nalgene compositing container (see SOP No. 2334.6 for tubing and container cleaning)
Volatile Organics <sup>1</sup>	Glass, Stainless steel (not solvent rinsed)
<b>B. Non-Aqueous Product Samples</b>	
All parameters	Glass

<sup>1</sup>An intermediate sample collection device is not recommended for this parameter, but, if one is necessary, care must be taken to insure that the device has not been solvent rinsed.

<sup>2</sup>An intermediate container is not recommended for this parameter; therefore, every effort must be made to collect the sample directly into the sample container.



Appendix B  
Example Field Sampling Forms



# Sample Collection Field Sheet

US EPA Region 7  
Kansas City, KS

ASR Number: 2567    Sample Number: 22    QC Code: \_\_\_\_    Matrix: Solid    Tag ID: 2567-22-\_\_\_\_

**Project ID:** JC07LT03    **Project Manager:** John Cook  
**Project Desc:** Madison County Mines - OU#3 Site  
**City:** Fredericktown    **State:** Missouri  
**Program:** Superfund  
**Site Name:** MADISON COUNTY MINES - MADWIDE    **Site ID:** 07LT    **Site OU:** 03

**Location Desc:** \_\_\_\_\_

**External Sample Number:** RSCPLA-14324

**Expected Conc:** \_\_\_\_\_ (or Circle One: Low Medium High)    **Date**    **Time(24 hr)**

**Latitude:** \_\_\_\_\_

**Sample Collection: Start:** 1/1 \_\_\_\_\_

**Longitude:** \_\_\_\_\_

**End:** 2/5/05 \_\_\_\_\_

## Laboratory Analyses:

Container	Preservative	Holding Time	Analysis
1 - 8 oz glass	4 Deg C	180 Days	1 Mercury in Soil or Sediment
1 - 8 oz glass	4 Deg C	180 Days	1 Metals in Solids by ICP

## Sample Comments:

(N/A)

**Sample Collected By:** \_\_\_\_\_




**CHAIN OF CUSTODY RECORD  
ENVIRONMENTAL PROTECTION AGENCY REGION VII**

ACTIVITY LEADER(Print) <b>John Cook</b>		NAME OF SURVEY OR ACTIVITY <b>FREDERICKTOWN LEAD</b>		DATE OF COLLECTION <b>17</b> DAY <b>03</b> MONTH <b>05</b> YEAR		SHEET <b>1</b> of <b>11</b>					
CONTENTS OF SHIPMENT <b>SOIL SAMPLES</b>											
SAMPLE NUMBER	TYPE OF CONTAINERS				SAMPLED MEDIA				RECEIVING LABORATORY REMARKS/OTHER INFORMATION (condition of samples upon receipt, other sample numbers, etc.)		
	CUBITAINER	BOTTLE	BOTTLE	BOTTLE	VOA SET (2 VIALS EA)	water	soil	sediment		dust	other
	NUMBERS OF CONTAINERS PER SAMPLE NUMBER										
2567-1		X					X				
2567-2											
2567-3											
2567-4											
2567-5											
2567-6											
2567-7											
2567-8											
2567-9											
2567-10											
2567-11											
2567-12											
2567-13											
2567-14											
2567-15											
2567-16											
2567-17											
2567-18											
2567-19											
2567-20											
2567-21											
2567-22											
2567-23											
2567-24		X					X				

DESCRIPTION OF SHIPMENT	MODE OF SHIPMENT
_____ PIECE(S) CONSISTING OF _____ BOX(ES) <input checked="" type="checkbox"/> ICE CHEST(S); OTHER _____	_____ COMMERCIAL CARRIER: _____ <input checked="" type="checkbox"/> COURIER <input checked="" type="checkbox"/> SAMPLER CONVEYED (SHIPPING DOCUMENT NUMBER) _____

PERSONNEL CUSTODY RECORD			
RELINQUISHED BY (SAMPLER) <i>[Signature]</i>	DATE <b>3/17/05</b>	TIME <b>1000</b>	RECEIVED BY
<input checked="" type="checkbox"/> SEALED <input type="checkbox"/> UNSEALED	<input checked="" type="checkbox"/> SEALED		<input type="checkbox"/> SEALED <input type="checkbox"/> UNSEALED
REASON FOR CHANGE OF CUSTODY			
RELINQUISHED BY	DATE	TIME	RECEIVED BY
<input type="checkbox"/> SEALED <input type="checkbox"/> UNSEALED			<input type="checkbox"/> SEALED <input type="checkbox"/> UNSEALED
REASON FOR CHANGE OF CUSTODY			
RELINQUISHED BY	DATE	TIME	RECEIVED BY
<input type="checkbox"/> SEALED <input type="checkbox"/> UNSEALED			<input type="checkbox"/> SEALED <input type="checkbox"/> UNSEALED
REASON FOR CHANGE OF CUSTODY			



 <b>UNITED STATES ENVIRONMENTAL PROTECTION AGENCY OFFICIAL SAMPLE SEAL</b>	<b>SAMPLE NO.</b>	<b>DATE</b>	<b>SEAL BROKEN BY</b>	<b>DATE</b>
	<b>SIGNATURE</b>			
	<b>PRINT NAME AND TITLE</b> <i>(Inspector, Analyst or Technician)</i>			

EPA FORM  
7500-2(R7-76)

Example Custody Seal





Pro ID: JC07LT30TH    **2909-6-**\_\_  
Matrix: Solid            1 TOC S.2A  
Cont: 8 oz glass  
#1  
Pres: 4 Deg C

Start: \_\_/\_\_/\_\_    \_\_:\_\_

End: \_\_/\_\_/\_\_    \_\_:\_\_

Example Sample Label



**FedEx** Express **US Airbill**

FedEx  
Tracking  
Number

8512 5846 7255

1 **From** Please print and print hard.  
Date 02-28-06 Sender's FedEx Account Number 1060-3987-9  
Sender's Name Anita Buggins Phone (913) 458-2000  
Company BLACK&VEATCH SPECIAL PROJ CORP  
Address 6601 COLLEGE BLVD  
City OVERLAND PARK State KS ZIP 66211-1504

2 **Your Internal Billing Reference** 044711-0103 OPTIONAL

3 **To**  
Recipient's Name Nicole Roblez Phone ( )  
Company USEPA Region VII  
Recipient's Address 300 Minnesota Ave  
We cannot deliver to P.O. boxes or P.O. ZIP codes.

Address  
To request a package be held at a specific FedEx location, print FedEx address here.  
City Kansas City State KS ZIP 66101

**Try online shipping at fedex.com**

By using this Airbill you agree to the service conditions on the back of this Airbill and in our current Service Guide, including terms that limit our liability.  
**Questions? Visit our Web site at fedex.com**  
or call 1.800.GoFedEx 1.800.463.3339.

0302613373

MUR23

0215

Sender's Copy

**4a Express Package Service**

Packages up to 150 lbs.  
\* To meet location

☒ FedEx Priority Overnight  
Next business morning\*  
☐ FedEx Standard Overnight  
Next business afternoon\*  
☐ FedEx First Overnight  
Earliest next business morning  
delivery to select locations\*  
☐ FedEx 2Day  
Second business day\*  
☐ FedEx Express Saver  
Third business day\*  
FedEx Envelope rate not available. Minimum charge: One-pound rate.

**4b Express Freight Service**

Packages over 150 lbs.  
\*\* To meet location

☐ FedEx 1Day Freight\*  
Next business day\*\*  
☐ FedEx 2Day Freight  
Second business day\*\*  
☐ FedEx 3Day Freight  
Third business day\*\*

\* Call for Confirmation.

**5 Packaging**

\* Declared value limit \$500

☐ FedEx Envelope\*  
☐ FedEx Pak\*  
Includes FedEx Small Pak,  
FedEx Large Pak, and FedEx Sturdy Pak.  
☐ FedEx Box  
☐ FedEx Tube  
☒ Other

**6 Special Handling**

Include FedEx address in Section 3.

☐ **SATURDAY Delivery**  
Available ONLY for  
FedEx Priority Overnight, FedEx 2Day,  
FedEx 10Day Freight, and FedEx 3Day  
Freight to select ZIP codes.  
☐ **HOLD Weekday**  
at FedEx Location  
NOT Available for  
FedEx First Overnight  
☐ **HOLD Saturday**  
at FedEx Location  
Available ONLY for  
FedEx Priority Overnight and  
FedEx 2Day to select locations

Does this shipment contain dangerous goods?  
One box must be checked.  
☒ No ☐ Yes  
As per attached Shipper's Declaration  
☐ Yes  
Shipper's Declaration  
not required  
☐ Dry Ice  
Dry Ice, 9, UN 1845  
☐ Cargo Aircraft Only  
Dangerous goods (including Dry Ice) cannot be shipped in FedEx packaging.

**7 Payment** Bill to:

Enter FedEx Acct. No. or Credit Card No. below.

☒ Sender  
Acct. No. in Section  
7a will be billed.  
☐ Recipient  
☐ Third Party  
☐ Credit Card  
☐ Cash/Check

FedEx Acct. No.  
Credit Card No. Exp. Date  
Total Packages 1 Total Weight            Total Declared Value\* \$            .00  
FedEx Use Only  
\*Our liability is limited to \$100 unless you declare a higher value. See back for details.

**8 Sign to Authorize Delivery Without a Signature**

By signing you authorize us to deliver this shipment without obtaining a signature and agree to indemnify and hold us harmless from any resulting claims.

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Field Sheet  
Madison County Mines OU5 RD

Date: \_\_\_\_\_ Time: \_\_\_\_\_ Samplers: \_\_\_\_\_

Sample Location: \_\_\_\_\_

Sample Number: \_\_\_\_\_

Coordinates: \_\_\_\_\_ Northing \_\_\_\_\_ Easting

Sample Type: \_\_\_\_\_ Soil; \_\_\_\_\_ Flood Plain Soil; \_\_\_\_\_ Sediment; \_\_\_\_\_ Surface Water; \_\_\_\_\_ Ground Water

Type of Analysis: \_\_\_\_\_ XRF; \_\_\_\_\_ Laboratory

Sample Collection Method: \_\_\_\_\_ Pump \_\_\_\_\_ Scoop/Spoon \_\_\_\_\_ Backhoe (test pit) \_\_\_\_\_  
\_\_\_\_\_ Direct Fill

Field Measurements: \_\_\_\_\_ pH \_\_\_\_\_ Temperature (°C) \_\_\_\_\_ SP Conductivity (µS)

Lab analysis: \_\_\_\_\_ RCRA Metals \_\_\_\_\_ Lead \_\_\_\_\_ WQ Parameters

Water Containers Filled: \_\_\_\_\_ Total Metals \_\_\_\_\_ Dissolved Metals \_\_\_\_\_ WQ Parameters

XRF Analysis: \_\_\_\_\_ Lead

QC Samples Collected:

- Duplicate \_\_\_\_\_
- MS/MSD \_\_\_\_\_
- Rinsate Number: \_\_\_\_\_